ISBS
Institut für Straßenwesen
TU Braunschweig

Dynamic Shear Rheometer Testing of Asphalt Binder using 4 mm parallel plate geometry at low temperature

2nd Interlaboratory Study

including test results of 16 participating laboratories from Austria, Belgium, Czech Republic, France, Germany, Italy, Lithuania, Poland, and Sweden

Final Report

by

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1 Motivation

This study is intended to support the development of a unified test protocol for rheological characterization of asphalt binder at low temperature by means of the Dynamic Shear Rheometer (DSR) and the 4 mm parallel plate geometry, in relation to the draft European standard prEN 14770.

This study follows a first interlaboratory study (ILS) on using DSR 4 mm parallel plate geometry in 2019. While during the first ILS different individual test protocols of the participants were screened and compared, this second ILS was based on the use of a unique pre-defined test protocol. From always three test replicates statistical analysis was performed and test repeatability and reproducibility were determined.

2 Materials

All participants used the same two asphalt binders as used in ILS from 2019, i. e. plain asphalt binder (50/70), and polymer modified asphalt binder (25/55-55 A).

Table 1 shows softening point ring-and-ball and needle penetration for these binders. More detailed information on these binders can be found in the final report of the ILS from 2019 (Büchner et al., 2020b).

Table 1: Asphalt binders of the types 50/70 and 25/55-55 A (Büchner et al., 2020b)

Test Method		50/70	25/55-55 A
Softening Point Ring-and-Ball (EN 1426, 2015)	[°C]	51.3	58.0
Needle Penetration (EN 1427,2015)	[1/10mm]	64	46

3 Test protocol

All participants received the following instructions to perform the DSR tests for both binders according to a predefined test protocol (per email from May 14th, 2020): 4 mm DSR plate-plate geometry; temperature-frequency sweep (T-f-sweep) test considering threefold test repetition; test temperatures of 0, -10, -20, and -30 °C (if possible); test frequencies of 0.1; 1.0; 1.59 and 10 Hz. Test sequences for DSR softwares “rSpace” (Netzsch) and “RheoCompass” (Anton Paar) were provided. A unique Excel file sheet was provided to the participants to collect information on the used equipment, sample preparation, specimen conditioning, test parameters and test results (complex shear modulus G* and phase angle δ), as well as on any further individual detail.

The following instructions were given in regard to sample preparation and conditioning, and to the test parameters:

Sample preparation:

- A sub sample (~ 20 g) of each binder (50/70 and 25/55-55) is heated in an oven at 150 °C for 60 minutes.

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- The homogenized, fluid asphalt binder is filled in a silicone mold with a preferred diameter of 4 mm, however, molds with a diameter of 8 mm or sheet materials can also be used.
- The specimen is stored covered, for a duration of 2 to 72 h for 50/70 and of 12 to 72 h for 25/55-55 (according to EN 14770, 2012) .
- Before starting the test, the zero gap is set with both parallel plates at ambient temperature.
- The sample installation temperature is 50 °C for 50/70, and 60 °C for 25/55-55. These temperatures are held constant for 5 minutes to reach an equilibrium state before mounting the specimen. It is important that both geometries are heated to the according installation temperature.
- After inserting the specimen, the gap is set to a width of 2.1 mm (5 % higher than the final gap) for trimming the specimen which is done with a heated (~ 90 °C) stainless steel tool after waiting 30 seconds for the specimen to become sufficiently fluid.
- The final gap is set to a width of 2.0 mm.

Specimen conditioning:

- The temperature-frequency-sweep (T-f-Sweep) are run at test temperatures of 0, -10, -20 and -30 °C as well as at frequencies of 0.1, 1.0, 1.59 and 10 Hz. The test starts at 0 °C and a decreasing trend for subsequent test temperatures is selected. To ensure a mechanical and thermal equilibrium within the specimen, the time for reaching the equilibrium state is set to 15 min at 0 °C and at -10 °C, 20 min at -20 °C, and 30 min at -30 °C.
- For each test temperature, the normal force is kept zero during the temperature conditioning by using gap compensation. During testing, the gap is kept constant by using the gap-controlled testing mode of the DSR.

Test parameters:

- Using strain-controlled oscillation mode, the applied shear strain is 0.1 % for the test temperatures 0, -10 and -20 °C, and 0.05 % for a test temperature of-30 °C.
- The measurement duration for each data point is pre-defined based on the frequency: 120 seconds for 0.1 Hz, 20 seconds for 1.0 Hz, and 10 seconds for 1.59 as well as for 10 Hz.
- Each participant performs three repetitions of the T-f-Sweep tests for both binders.
- It is important that the compliance correction is applied to the results. Therefore, each laboratory has to check and report the instruments' shear compliance values.

4 Participation

16 laboratories participated in this interlaboratory study, covering Alma Petroli (Italy), Anton Paar (Germany), Brno University of Technology (Czech Republic), COLAS (France), Gdansk University of Technology (Poland), Nynas (Sweden, Belgium), Road and Bridge

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Research Institute (Poland), Ruhr-Universität Bochum (Germany), RWTH Aachen (Germany), Spie Batignolles Malet (France), TU Braunschweig (Germany), TU Wien (Austria), Unipetrol (Czech Republic), Vialab (Czech Republic), and Vilnius Gediminas Technical University (Lithuania).

One participant provided three separate test results originating from three different DSR. A total number of 18 data sets was received, between July 29th, 2020 and September 18th, 2020.

5 Analysis

Anonymous laboratory numbers were assigned to the data sets. Each participant knows the laboratory-specific number, only.

General information on the testing equipment and the tests is summarized in Chapters 6.1 to 6.4. The Annex includes all rheological parameters reported.

Based on the resulting values of complex shear modulus and phase angle, the following parameters were calculated from all data sets:

- *cell mean y** of the three individual replicates from each laboratory,
- *general mean y*** of all test results from all laboratories after eventual statistical exclusion according to Cochran's and Grubbs' test,
- *standard deviation* of the general mean (in percent) after eventual statistical exclusion according to Cochran's and Grubbs' test,
- *Mandel's k* within laboratory consistency statistic,
- *Mandel's h* between laboratory consistency statistic,
- *repeatability* $r = 2.77 \cdot s_r$ in kPa or °, and in % from general mean,
- *reproducibility* $R = 2.77 \cdot s_R$ in kPa or °, and in % from general mean.

Two different numerical outlier identification techniques, the Cochran's test and the Grubbs' test, were used to identify possible stragglers or outliers according to ISO 5725-2 (2019).

Cochran's test was used for identifying the "within-laboratory variabilities". It was applied before the Grubbs' test, which is for identifying the "between-laboratory variability". All stragglers and outliers are reported in Chapter 9.

Mandel's k and h statistics are also covered in ISO 5725-2 (2019). These statistical techniques are suitable to analyze the graphical consistency of the data. Mandel's k value is indicating the "within-laboratory consistency" and Mandel's h value is indicating the "between-laboratory consistency". While Mandel's k results can only be positive, Mandel's h results can be positive or negative. Both statistics can be plotted separately for each test parameter.

As to Mandel's k statistics, for a laboratory having many large values it can be assumed that repeatability is lower compared to other laboratories.

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As to Mandel's h, there are various patterns possible. For example, for individual laboratories always positive or always negative values may be obtained. Usually, the number of laboratories with negative results is almost equal to the number of laboratories with positive values only.

According to ISO 5725-2 (2019) different limiting values (depending on the number of laboratories and the number of results per laboratory) can be calculated to evaluate consistency patterns. Detailed analysis of Mandel's k and Mandel's h values – both for a frequency of 1.59 Hz – is included in Chapters 6.5.1 and 6.5.2, respectively.

The characteristic values of repeatability r and reproducibility R are calculated following the procedure given in ISO 5725-2 (2019) based on repeatability variance s_r^2, between-laboratory variance s_L^2, and reproducibility variance s_R^2. The results for all temperature and frequency combinations for both materials are visualized in Chapter 6.5.3.

6 Test results

6.1 Test equipment

Information on test equipment used by the participants is provided in the following Tables.

Laboratory	Brand of DSR	Model of DSR	Year of purchase	DSR control software
1	Anton Paar	MCR 502	2017	RheoCompass 1.25, FW 5.01
2	Anton Paar	MCR 302	2016	RheoCompass 1.25
3	Anton Paar	MCR 702 Multidrive	2019	RheoCompass 1.24
4	Anton Paar	MCR-101	2008	RHEOPLUS/32 V2.81
5	Netzsch	Kinexus DSR+	2015	rSpace 1.75
6	Anton Paar	Smart Pave 102	2019	RheoCompass 1.25.373
7	Anton Paar	MCR 702	2019	RheoCompass 1.25
8	Netzsch	Kinexus pro +	2014	rSpace 1.75
9	Netzsch	Kinexus pro	2013	rSpace 1.75
10	Anton Paar	EC-Twist 502	2014	RheoCompass 1.25
11	Anton Paar	MCR 302	2019	RheoCompass 1.24
12	TA Instruments	Discover HR 3	2015	Trios v4
13	Anton Paar	MCR 5002	2014	RheoCompass 1.20.493
14	Anton Paar	MCR Smart Pave 102	2018	RheoCompass 1.24
15	Netzsch	Kinexus PRO	2012	rSpace 1.76
16	Anton Paar	MCR 501	?	RheoCompass 1.23
17	Anton Paar	MCR 302	2019 (2020 delivery and in operation)	RheoCompass 1.25.355
18	Netzsch	Kinexus DSR	2018	rSpace 1.76.2398

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Laboratory	Temperature control system	Model of temperature control system
1	Peltier	H-PTD 200, P-PTD200/56/I
2	Peltier	H-PTD 200, P-PTD 200/56/I
3	Peltier	H-PTD 200 + P-PTD 200
4	Peltier	TU1=TEK150PA-CF (this is under Peltier) +H-PTD200- (this is the upper Peltier - was acquired 2018)
5	Peltier	-
6	Peltier	H-PTD 200
7	Peltier	H-PTD 200
8	Peltier	KNX2007
9	Peltier	KNX2007
10	Peltier	H-PTD 200
11	Peltier	H-PTD 200
12	liquid nitrogen (at low temperatures)	TA Environmental Test Chamber (ETC)
13	Peltier	H-PTD 200+P-PTD 200
14	Peltier	P-PTD200+H-PTD120
15	Peltier	KNX2007
16	Peltier	H-PTD 200
17	Peltier	P-PTD200/80/I + H-PTD200
18	Peltier	-

laboratory	Is an external heat exchanger used for counter cooling?	If YES, brand and model	If YES, liquid used for counter cooling	If YES, is the external heat exchanger controlled by the DSR?	If YES, is the external heat exchanger controlled manually?	If YES, counter cooling temperature
1	YES	CP-600F	Water-glycol mixture (60:40)	NO	YES	-10
2	YES	Viscotherm VT2	Antifreeze	YES	YES	-8
3	YES	Viscotherm VT2	Antifreeze	NO	YES	10
4	YES	Minichiller Petroplus	water -glycerol 50/50	NO	YES	-10
5	YES	Julabo CF41	Antifreeze G12+	YES	NO	-
6	YES	Julabo FP50	water/glycol	YES	NO	-
7	YES	Julabo FP50	Thermal g	YES	NO	-10
8	YES	Julabo CF41	Thermal g	YES	NO	-
9	YES	Julabo CF41	Thermal g	YES	NO	-
10	YES	Julabo FP50	Thermal g	YES	NO	-
11	YES	Anton Paar - Viscotherm VT 2	50 % Glycol - 50 % Water	YES	NO	-
12	NO	-	-	-	-	-
13	YES	Viscotherm VT2	Antifreeze	YES	NO	-
14	NO	-	-	-	-	-
15	YES	Julabo CF41	Fridex	YES	NO	-
16	YES	Anton Paar	Glycol	YES	NO	-
17	YES	Julabo FP50	Thermal g	YES	NO	-
18	YES	Julabo CF41	Fridex G48	YES	NO	-

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laboratory	Is the 4mm-geometry serrated?	Compliance of 4mm-geometry used
	(YES/NO)	[rad/Nm]
1	NO	0.016
2	NO	0.0258
3	YES	0.0017
4	NO	YES
5	YES	0.002357
6	YES	0.016
7	YES	0.02
8	YES	0.014
9	YES	0.014
10	NO	0.0017
11	NO	0.016
12	NO	-
13	NO	0.016
14	NO	0.0017
15	NO	0.002356
16	NO	0.0017
17	NO	0.016
18	YES	0.002356

Comments
Lab. 4 All information from ILS 4 mm 2019, laboratory did not provide current information.
Lab. 12 I will send the compliance later
Lab. 14 Mentioned procedure from specification tab was applied to test successfully.

6.2 Sample preparation

Information on sample preparation is provided in the following Tables.

laboratory	Where the entire containers with the test material heated in an oven during the first ILS 2019?	Specify material of mold or sheet	Specify shape of the specimen
1	YES	Silicone	Cylinder with diameter of 4 mm, 2.5 mm thickness and convex top
2	NO	Silicone	cylindrical
3	-	Sheet	Droplet
4	YES	Silicon molds	2.5 mm height, 4.2 mm diameter
5	YES	Silicone	circular cross mould, diameter ≈ 3mm
6	YES	Silicone	Cylinder
7	NO	Silicone	Cylindrical
8	NO	Silicone	Cylindrical
9	NO	Silicone	Cylindrical
10	YES	Silicone	standard DSR-Specimen
11	YES	Silicone mould	Circular, with a diameter of 4mm
12	YES	Silicone mould	Cylindrical
13	YES	Silicone mould	Cylinder
14	NO	Silicone mould	Cylinder / ring shape
15	YES	two-component silicone rubber (Lukopren N 1522)	Cylindrical
16	YES	mould	cylinder
17	NO	Silicone	Cylindrical
18	YES	silicone rubber, hand made	cylindrical, roughly 5 mm in diameter (a lot of excessive material), need to have smaller next one

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laboratory	Storage time before demolding and testing for 50/70	Storage time before demolding and testing for 25/-55-55
	[min]	[min]
1	135 min, 305 min, 470 min	985 min, 1155 min, 1335 min
2	1020-1260	2580-2970
3	120	1440
4	15, 115, 190	1140, 1260, 1360
5	120	720
6	120	1440
7	960	960
8	960	960
9	960	960
10	120	1440
11	720	720
12	15	15
13	120/1233/1367	1050/1170/1325
14	720	720
15	1200	1200
16	1080	1080
17	120 – 1560	720 - 2160
18	180 min	180 min

Comments

Lab. 1 All samples for one material were prepared at once. Therefore 3 storage times for each material are listed.

Lab. 2 specimens made of 50/70 were demolded after 17, 19 and 21 hours;
specimens made of PMB 25/55-55 were demolded after 43, 45.5 and 49.5 hours.

Lab. 3 The storage times followed according to EN 14770

Lab. 14 Both asphalts were demolded after 12h.

Lab. 17 (1) - We didn't join ILS 2019. Samples have been shipped to our lab in July 2020. (2) To prepare the sample small quantity of bitumen was transferred to a metallic pan and heated with help of a heat gun. The heating of the bitumen was conducted until it is fluid enough to pour into the silicon molds (diameter of 8 mm). Usually, it was prepared four samples during the morning. Then, after 2 or 12 hours the sample was demolded from the silicon mold and the first bitumen sample tested. The remaining samples were then tested on the same day or at maximum on the next day.

Lab. 18 A specific mold from the manufacturer for the sample preparation in the DSR was used last year. In this case, the height of the sample was 3 mm. This year a 2 mm gap was required to use. Therefore, the tool could have not been used. As a result, a manual trimming of the sample was needed. Sample preparation plays a key role in getting the results with a good repeatability. I think that repeatability might be hindered by not using a specific mold (not matter which one, but the same for all the participants). Storage time differed from sample to sample because one sequence is about 3 hours. There cannot be only one time because we poured more analytical samples at one time. Is there any data which proves that during the time span from 2 hours to 72 hours the samples remain representative? If so, could you please share those? Temperature 90 °C was not sufficient for cutting the bitumen sample. We had to heat it up to higher temperatures.

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6.3 Sample conditioning and test parameters

Information on sample conditioning and test parameters is provided in the following Table.

laboratory	Has gas/air been used to prevent ice formation at the interface between the geometry and the temperature hood?	If YES, specify the type of gas/air and the volume flow
	(YES/NO)	[min]
1	YES	180 LN/h
2	YES	air, 200 lN/h
3	YES	air, 200 lN/h
4	YES	dry air/ 200l/h
5	NO	-
6	YES	air
7	YES	Nitrogen 99,999 %, 2 L/min
8	YES	Nitrogen 99,999 %, 2 L/min
9	YES	Nitrogen 99,999 %, 2 L/min
10	NO	-
11	YES	Pressurized air - 100 LN/h
12	YES	nitrogen, volume flow depends on temperature, increases for lower temp.
13	YES	comprised air at 200l/h
14	NO	-
15	NO	-
16	NO	-
17	YES	Dry Air (1) / 200 [l/h]
18	YES	dried air (pressure 2,0 - 2,5 bar)

Comments

Lab. 14 This action wasn't needed

Lab. 17 (1) - DRYPOINT® M compressed-air membrane dryer

Lab. 18 Ice formation was detected although equipment for using dry air installed by a manufacturer's representative was put into action. It is strange that there is hardly any contraction on the sample during conditioning. I would suppose that the gap will be smaller and smaller as temperature decreases. In the sequence pretty much, the same gap was at the beginning as in the end. Sometimes the gap was a little bit bigger in the end than at the beginning. Do you have any explanation for that? The normal force was kept zero all the time.

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6.4 Rheological parameters

Information on rheological parameters is provided in the following Table.

laboratory	Have additional test results beyond the below stated been discarded? (YES/NO)	If YES, why?
1	YES	1 additional test for the 50/70. Gap was not fully filled
2	NO	-
3	NO	-
4	NO	-
5	NO	-
6	NO	-
7	NO	-
8	NO	-
9	NO	-
10	NO	-
11	YES	Some test were beyond the reproducibly limit
12	NO	-
13	NO	-
14	YES	"Only one per asphalt to check spindle calibration and results performed throughout the received Rheocompass file. "
15	NO	-
16	NO	-
17	YES	No previous experiences with the 4mm - geometry
18	NO	-

6.5 Statistical analysis

This chapter summarizes the results of the statistical analysis in form of conclusive graphs. First, Mandel's k and Mandel's k values are presented to evaluate the graphical consistency of the test results at 1.59 Hz. Graphical results for other frequencies are very similar and are not included in this report. For Mandel's k and Mandel's outliers according to Grubb's and Cochran's tests are included in the Figures.

Finally, test repeatability and reproducibility values for all temperatures and frequencies are evaluated (by excluding Grubb's and Cochran's outliers).

6.5.1 Mandel's k consistency analysis

As mentioned in Chapter 5, Mandel's k is used to evaluate the within-laboratory consistency, where high values are an indicator for poor repeatability compared to remaining laboratories.

Figure 1 represents Mandel's k values for the complex shear modulus of the 50/70 binder at a frequency of 1.59 Hz, including the different test temperatures of 0, -10, -20 and -30 °C.

It can be observed that some laboratories like 3 and 15 have a better repeatability with small Mandel's k values, while some laboratories like 4 and 12 have low repeatability with high Mandel's k values, also reaching the k limit according to ISO 5725-2 (2019). Most laboratories show comparable Mandel's k values over the temperatures tested. However, laboratory 12 has very inconsistent results with lowest repeatability of test results at -30 °C. Interestingly, the Mandel's k value at -30 °C is usually lower than for all other temperatures.

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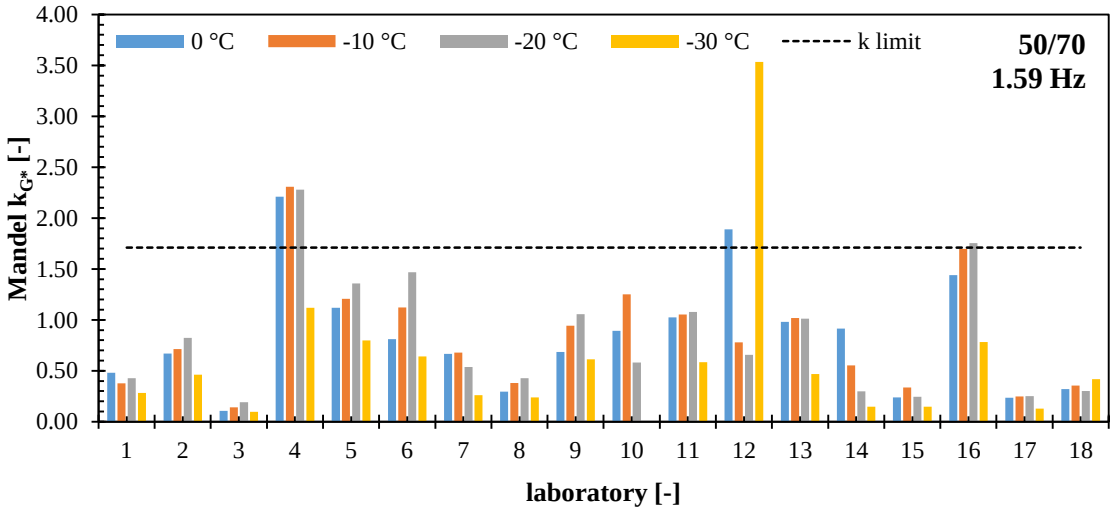

Figure 1. Mandel's k results for the complex shear modulus G* (50/70 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 2 represents Mandel's k values for the 25/55-55 binder. The results are very similar compared to Figure 1, again with laboratory 4 and 12 reaching the k limit.

It is recommended to these laboratories to review their testing techniques in order to improve repeatability of test results (for laboratory 12 especially at -30 °C).

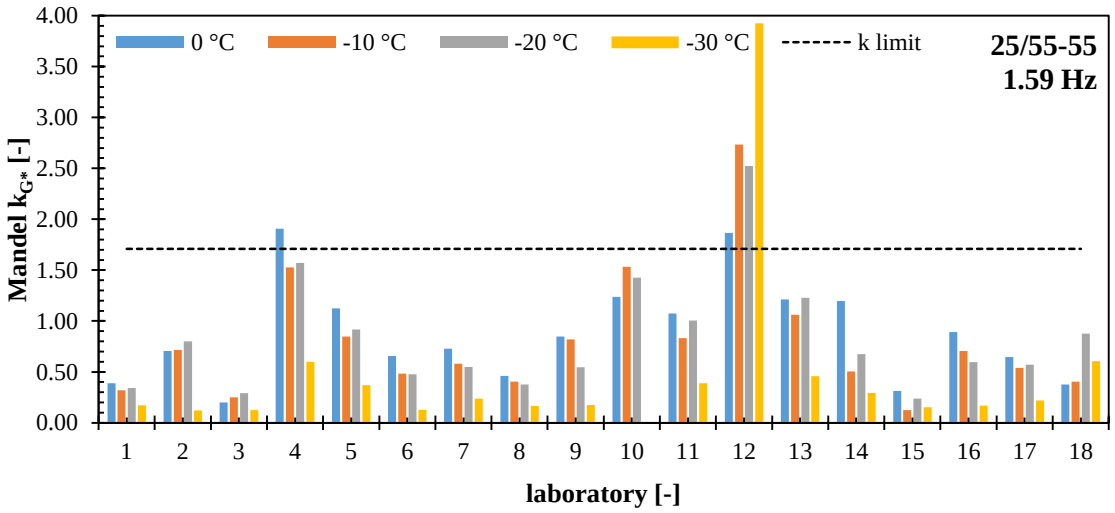

Figure 2. Mandel's k results for the complex shear modulus G* (25/55-55 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 3 represents Mandel's k values for the phase angle of the 50/70 binder at a frequency of 1.59 Hz, including the different test temperatures of 0, -10, -20 and -30 °C. It can be seen that the within-laboratory consistency varies largely between the different laboratories. Laboratories 8 and 9 show better repeatability than 7 and 12.

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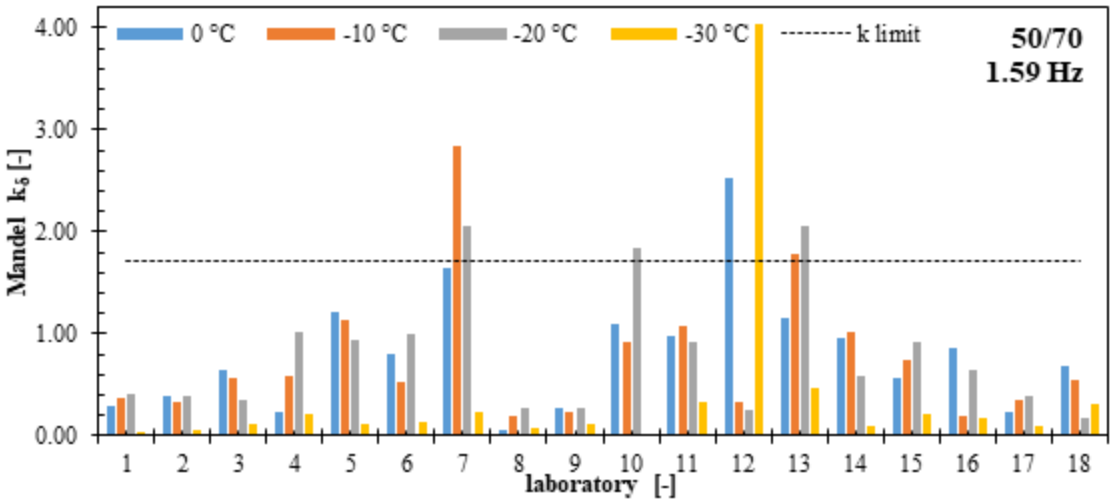

Figure 3. Mandel's k results for the phase angle δ (50/70 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 4 represents Mandel's k values for the 25/55-55 binder. While repeatability of laboratory 12 is again peculiar, consistency varies slightly between the laboratories. Laboratory 7 shows best repeatability, while it is worst for laboratory 9.

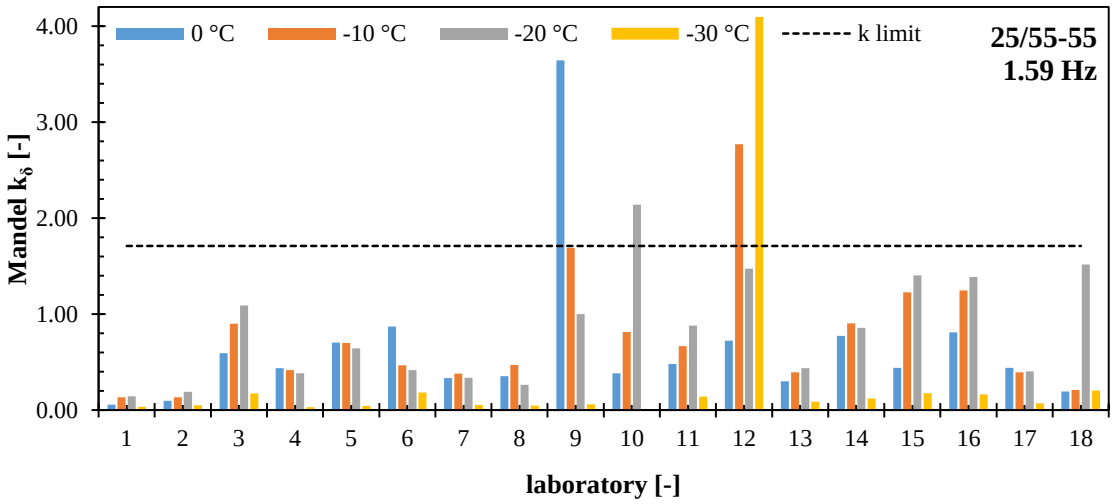

Figure 4. Mandel's k results for the phase angle δ (25/55-55 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

6.5.2 Mandel's h consistency analysis

Mandel's h value allows for evaluating the between-laboratory consistency of test results. High values indicate satisfying between-laboratory consistency compared to the general mean, and vice versa.

Figure 5 represents Mandel's k values for the complex shear modulus of the 50/70 binder at a frequency of 1.59 Hz, including the different test temperatures of 0, -10, -20 and -30 °C. First, it can be noted, that approximately the same number of laboratories show positive Mandel's h values as negative values, which is an expected pattern. At the same time, some

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laboratories are reaching the h limit of 1.88, indicating less consistency with other laboratories and a huge deviation of their test results from the general mean value. For example, laboratories 3, 5 and 12 (the latter only at -30 °C) show smallest values, and laboratory 14 shows highest values.

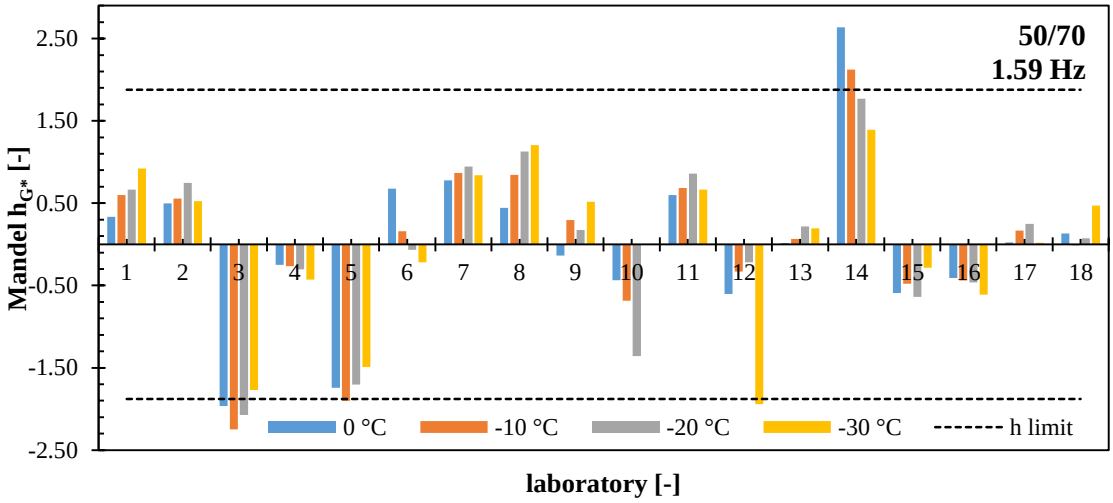

Figure 5. Mandel's h results for the complex shear modulus G* (50/70 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 6 represents Mandel's h values for the 25/55-55 binder. The results are very similar to the 50/70 binder (Figure 5), with the same laboratories reaching the k limit. This indicates, that there seems to be a systematic error related to determination of G* for some laboratories.

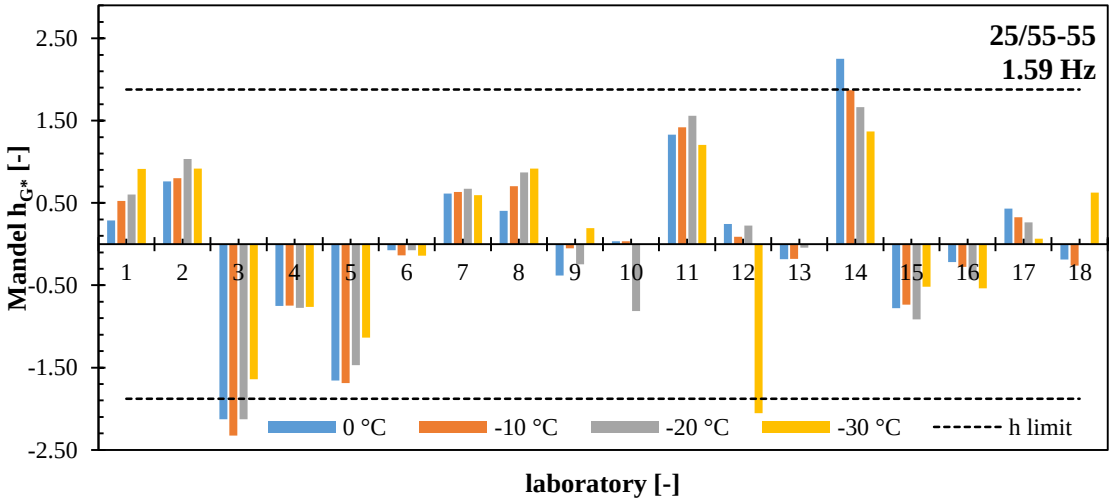

Figure 6. Mandel's h results for the complex shear modulus G* (25/55-55 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 6 represents Mandel's h values for the phase angle of the 50/70 binder at a frequency of 1.59 Hz, including the different test temperatures of 0, -10, -20 and -30 °C. The test results of laboratory 3 are highest, and laboratory 6 is almost reaching the h limit at a test temperature of 0 °C.

https://doi.org/10.24355/dbbs.084-202110271159-0

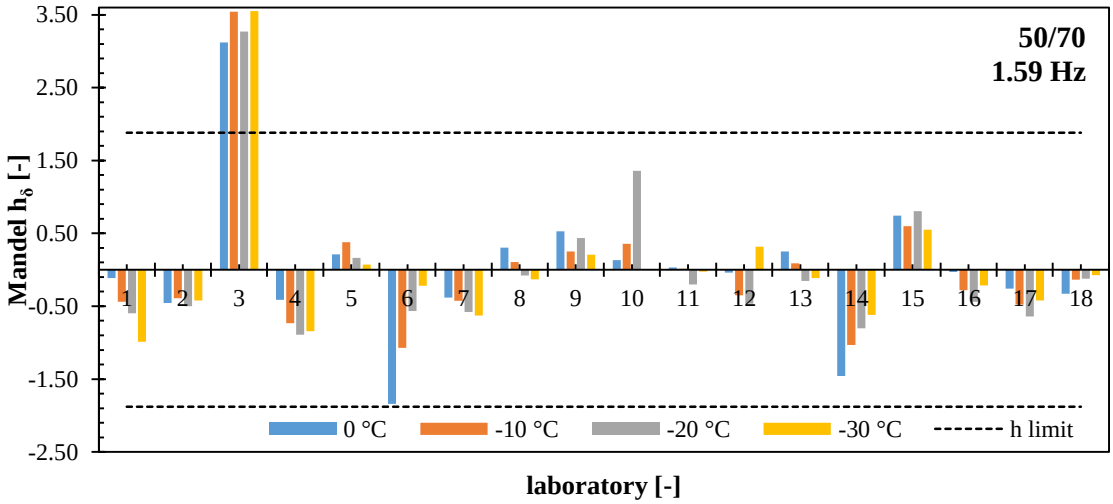

Figure 7. Mandel's h results for the phase angle δ (50/70 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

Figure 8 represents Mandel's k values for the 25/55-55 binder. The results are quite similar to the results obtained for the 50/70 binder (Figure 7). Again, laboratory 3 shows highest values. This indicates, that there is a systematic error related to measuring and/or determining the phase angle δ.

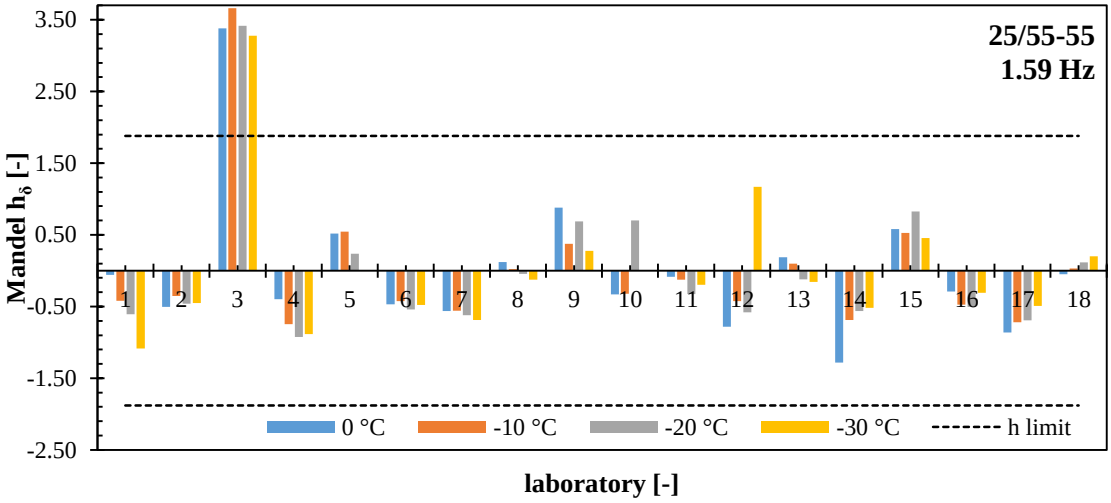

Figure 8. Mandel's h results for the phase angle δ (25/55-55 binder) at 1.59 Hz for all laboratories from 0 to -30 °C.

6.5.3 Repeatability and reproducibility

In this report, repeatability and reproducibility represent limiting values, calculated from multiplying the respective standard deviations with 2.77, indicating the 95 % confidence interval of test results. These values are also included as general DSR precision data in the upcoming prEN 14770 (currently under revision).

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In Figure 9, repeatability r and reproducibility R for obtaining the complex shear modulus (in % from the general mean value) are represented for both binders, including all temperature and frequency data sets. The following observations can be made:

- repeatability is in the range of 15 to 20 %, while the reproducibility is in the range of 65 to 95 %,
- repeatability and reproducibility of the 25/55-55 binder are always slightly better (showing smaller values) than of the 50/70 binder,
- repeatability values are similar for all temperatures and frequencies and seem to be independent from testing conditions,
- reproducibility values seem to be slightly dependent on the test frequency, with higher frequencies having a better repeatability,

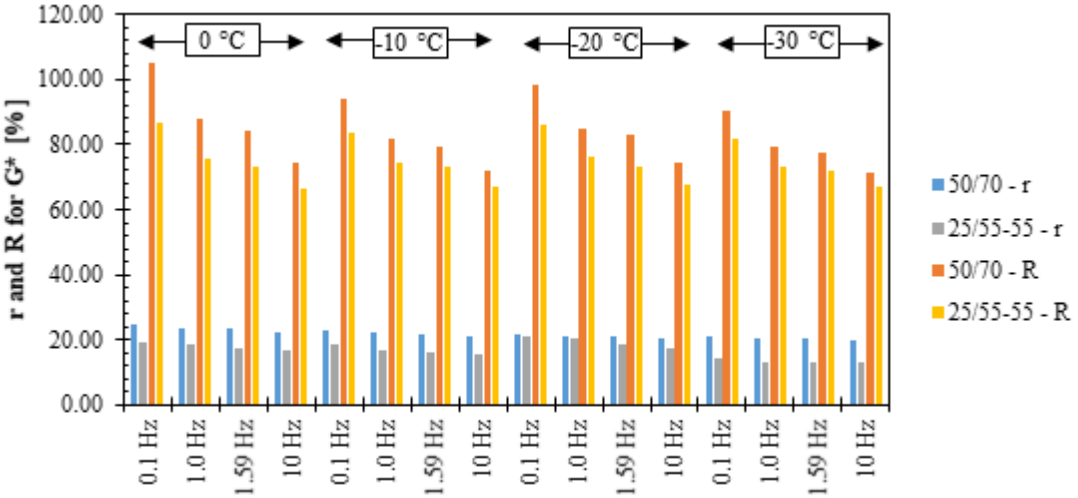

Figure 9. Repeatability r and reproducibility R from the general mean in percent for the complex shear modulus G* for all frequencies and temperatures.

In Figure 10, repeatability r and reproducibility R for obtaining the phase angle (in % from the general mean value) are represented for both binders, including all temperature and frequency combinations. It can be seen that the precision of test results worsens with decreasing temperature. However, representation of the phase angle in % is misleading, because the precision of determining the phase angle is independent from the absolute value (see also Büchner et al. (2020a)). This is also the reason, why during revision of prEN 14770 it was decided to evaluate precision of the phase in absolute values instead.

https://doi.org/10.24355/dbbs.084-202110271159-0

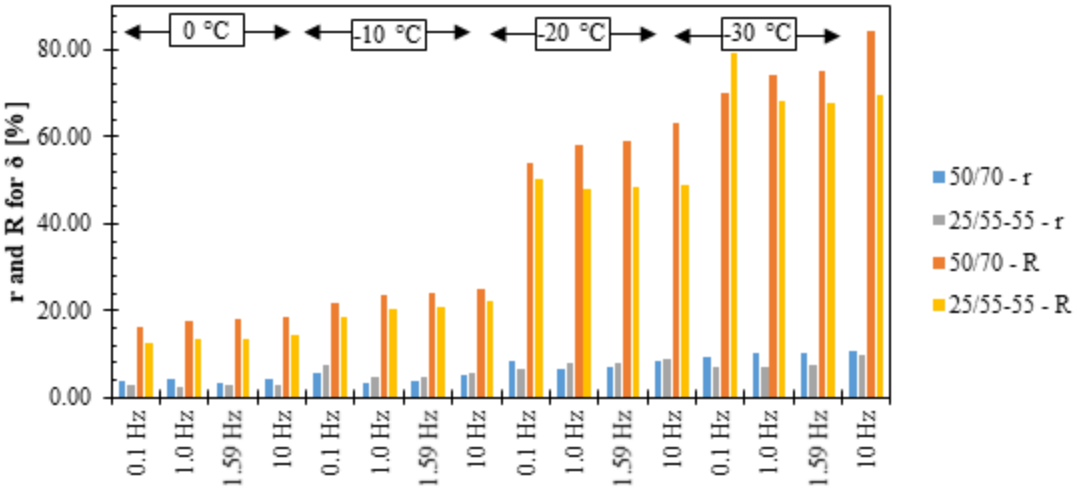

Figure 10. Repeatability r and reproducibility R from the general mean in percent for the phase angle δ for all frequencies and temperatures.

Figure 11 represents repeatability r and reproducibility R for obtaining the phase angle in absolute values. The following observations can be made:

- repeatability is in the range of 0.5 to 1.5 %, while the reproducibility is mainly in the range of 3 to 7 %,
- repeatability and reproducibility of the 25/55-55 binder are always slightly better (showing smaller values) than of the 50/70 binder,
- repeatability values are slightly smaller for lower temperatures and higher frequencies,
- reproducibility values seem to be dependent on the test frequency, with higher frequencies having a better repeatability; for -20 and -30 the reproducibility, especially at 0.1 Hz is detrimental as the general mean value is in the range of 10 ° while reproducibility limit is also around 10°.

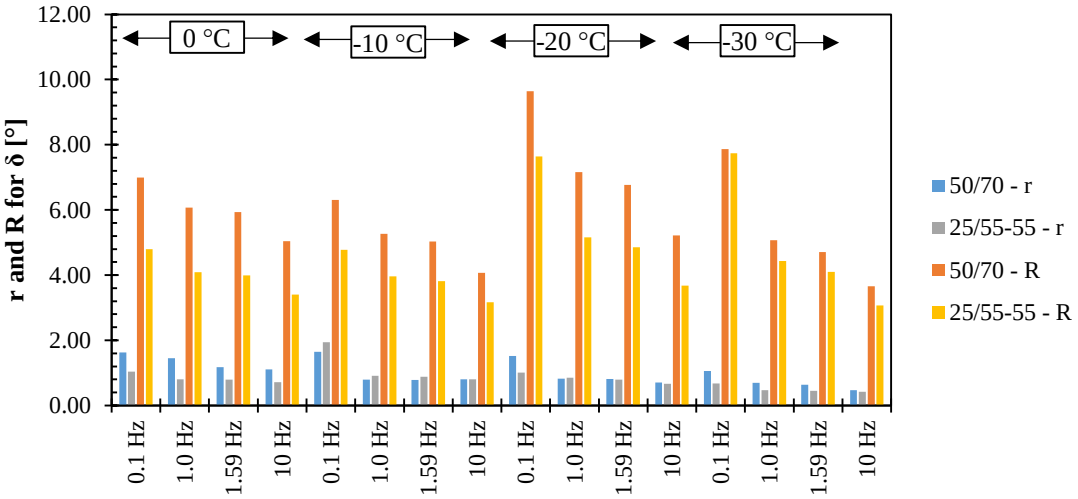

Figure 11. Repeatability r and reproducibility in absolute values for the phase angle δ for all frequencies and temperatures.

https://doi.org/10.24355/dbbs.084-202110271159-0

In Table 2, repeatability and reproducibility values are summarized and compared to the values from the first ILS in 2019. The table also includes the precision values for 8 and 25 mm geometries included in the current draft of prEN 14770 (2021). It can be seen, that the repeatability values of 4 mm DSR are very similar to the values given in prEN 14770, while the reproducibility values of 4 mm are much higher (indicating a poor reproducibility).

Table 2: Comparison of repeatability and reproducibility values

	ILS 4 mm (2020) given protocol	**ILS 4 mm (2019) own protocol**	**prEN 14770 (2021) for 8 and 25 mm**
Repeatability r for G* in % from mean value	15 to 20 %	15 to 20 %	15 %
Reproducibility R for G* in % from mean value	65 to 95 %	50 to 75 %	30 %
Repeatability r for δ in °	0.5 to 1.5°	1.5 to 2.5°	2 °
Reproducibility R for δ in °	3 to 7°	3 to 6 °	4 °

7 Summary and conclusions

This second interlaboratory study includes 16 participants to investigate the rheological low temperature behavior of asphalt binders using the 4 mm parallel plate geometry of the Dynamic Shear Rheometer (DSR). Two asphalt binders were considered, i. e. plain binder of the type 50/70, and polymer modified asphalt binder of the type 25/55-55 A.

Each participant performed temperature-frequency-sweep tests from 0 to -30 °C and from 0.1 to 10 Hz following a predefined test protocol, with the objective to improve the reproducibility of test results and complementing the first interlaboratory study from 2019 (Büchner et al., 2020a). All participants followed detailed instructions for sample preparation, specimen conditioning and testing.

18 individual test results were received, as one of the laboratories provided three test results from different test instruments. Along with the test results (complex shear modulus G* and phase angle δ), information on the used equipment was collected.

Statistical analysis was carried out according to ISO 5725-2 (2019). Numerical outliers were determined according to Cochran's and Grubbs' standard tests, consistency of test results was analyzed with Mandel's k and h statistics, and finally, repeatability ($r = 2{,}8 \cdot s_r$) and reproducibility ($R = 2{,}8 \cdot s_R$) were calculated and evaluated with the following outcomes:

- some provided test results were implausible or inconsistent and were excluded after performing the Cochran's or Grubbs' tests;
- with Mandel's k statistics the "within-laboratory consistency" was analyzed and conspicuities for laboratories 3, 7, 8 and 12 were identified;

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- with Mandel's h statistics the "between-laboratory consistency" was analyzed and conspicuities for laboratories 3, 5, 12 and 14 were identified;
- the repeatability of the phase angle was found in the range of 0.5 to 1.5° and for the complex shear modulus in the range of 15 to 20 % (in percent from general mean value), confirming results of the first interlaboratory study and almost matching precision values of prEN 14470;
- the reproducibility of the phase angle was found in the range of 3 to 6 °, also confirming results of the first interlaboratory study and almost matching precision values of prEN 14770;
- the reproducibility of the complex shear modulus was found in the range of 65 to 95 %.

It can be concluded that the objective to improve the reproducibility by using a pre-defined test protocol to allow testing under reproducibility conditions could not be achieved at this stage. It remains unclear why reproducibility was not improved despite detailed testing instructions. However, it seems that the different devices used, and the individual experience of laboratory employees (significantly affecting the trimming of the specimen) were the reason for the low reproducibility. Another factor might be some kind of aging effect of the binder, as some (but not all) participants heated the entire sample container instead of only the needed amount for testing during the first interlaboratory study.

This second interlaboratory study demonstrated the general applicability of the 4 mm parallel plate for low temperature asphalt binder testing due to the acceptable repeatability within the laboratories. However, further investigations are needed.

Acknowledgement

Technische Universität Braunschweig sincerely thanks all laboratories for participating in this interlaboratory study, i. e. Alma Petroli (Italy), Anton Paar (Germany), Brno University of Technology (Czech Republic), COLAS (France), Gdansk University of Technology (Poland), Nynas (Sweden, Belgium), Road and Bridge Research Institute (Poland), Ruhr-Universität Bochum (Germany), RWTH Aachen (Germany), Spie Batignolles Malet (France), TU Wien (Austria), Unipetrol (Czech Republic), Vialab (Czech Republic), and Vilnius Gediminas Technical University (Lithuania).

8 References

Büchner, J., Wistuba, M. P., Dasek, O., Staschkiewicz, M., Soenen, H., Zofka, A. & Remmler, T. 2020a. Interlaboratory Study on Low Temperature Asphalt Binder Testing Using Dynamic Shear Rheometer with 4 mm Diameter Parallel Plate Geometry. Road Materials and Pavement Design, Taylor & Francis. DOI: 10.1080/14680629.2020.1851291.

https://doi.org/10.24355/dbbs.084-202110271159-0

Büchner, J., Wistuba, M. P. & Klüter, I. 2020b. Asphalt Binder Testing using 4 mm parallel plate geometry of Dynamic Shear Rheometer - Interlaboratory Study including test results of 20 participating laboratories from Belgium, Czech Republic, Denmark, Finland, France, Germany, Italy, Lithuania, and Poland. Final Report, Technische Universität Braunschweig, Institut für Straßenwesen, Braunschweig.

EN 14770, 2012. Bitumen and bituminous binders – Determination of complex shear modulus and phase angle using a Dynamic Shear Rheometer (DSR). European Committee for Standardization (CEN), Brussels.

ISO 5725-2, 2019. Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method. International Organization for Standardization (ISO), Geneva, Switzerland.

https://doi.org/10.24355/dbbs.084-202110271159-0

9 ANNEX: Numerical results

9.1 Complex shear modulus G* for 50/70

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	21,570	22,730	23,300	22,533
2	23,820	23,490	25,800	24,370
3	6,298	6,289	6,169	6,252
4	23,290	19,151	15,671	19,371
5	11,900	8,940	7,918	9,586
6	30,088	30,711	28,441	29,747
7	27,420	24,780	26,190	26,130
8	21,879	22,812	22,035	22,242
9	19,499	17,383	18,730	18,537
10	17,340	18,760	16,200	17,433
11	21,620	24,860	25,230	23,903
12	15,864	13,951	21,312	17,042
13	17,975	19,585	21,911	19,824
14	44,020	43,350	39,110	42,160
15	16,044	15,070	15,390	15,501
16	18,290	19,580	15,400	17,757
17	21,380	20,530	20,630	20,847
18	20,897	20,967	22,181	21,348

analytical exclusions:
-

statistical exclusions:
-

after exclusion:
number of laboratories p: 18
general mean y**: 20,810
standard deviation s: 7,673

notes:
Lab. 14 straggler after Grubbs Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	114,100	117,200	119,300	116,867
2	114,500	110,800	121,100	115,467
3	23,620	24,490	23,740	23,950
4	115,200	94,331	76,990	95,507
5	51,500	42,300	34,530	42,777
6	117,567	121,019	104,089	114,225
7	132,400	119,500	126,100	126,000
8	115,342	122,079	117,673	118,365
9	107,641	94,371	101,897	101,303
10	81,380	74,900	64,430	73,570
11	103,400	119,100	117,600	113,367
12	92,630	85,174	96,753	91,519
13	87,148	94,836	106,316	96,100
14	178,200	174,400	163,600	172,067
15	78,395	72,782	77,703	76,293
16	87,360	97,520	72,560	85,813
17	107,800	103,900	103,600	105,100
18	95,224	99,642	101,644	98,836

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 98,174
standard deviation s: 32,274

notes:
Lab. 4 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	351,000	358,000	368,100	359,033
2	360,300	344,900	380,800	362,000
3	69,480	73,640	70,120	71,080
4	341,600	284,873	230,166	285,546
5	162,000	141,000	113,959	138,986
6	311,212	322,064	271,936	301,737
7	401,200	375,900	379,800	385,633
8	366,058	384,922	375,619	375,533
9	299,033	257,024	272,896	276,318
10	149,000	142,600	130,100	140,567
11	328,500	377,000	361,600	355,700
12	280,308	261,951	290,408	277,556
13	277,781	294,380	331,828	301,330
14	485,300	474,200	460,900	473,467
15	206,162	195,737	204,607	202,168
16	253,500	294,500	214,600	254,200
17	332,500	322,000	319,100	324,533
18	285,964	297,305	304,343	295,871

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 287,848
standard deviation s: 99,474

notes:
Lab. 4 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	669,600	677,600	707,200	684,800
2	593,900	538,100	586,800	572,933
3	130,400	136,100	130,600	132,367
4	533,100	462,064	359,312	451,492
5	294,000	258,000	198,279	250,093
6	474,725	483,417	398,730	452,290
7	665,700	633,200	625,800	641,567
8	644,576	688,578	647,662	660,272
9	573,139	498,662	532,416	534,739
10				
11	514,000	601,000	578,500	564,500
12	63,260	515,398	67,804	215,487
13	468,969	493,458	548,060	503,496
14	745,700	730,100	716,100	730,633
15	401,719	383,688	403,066	396,158
16	388,200	437,700	335,600	387,167
17	508,200	491,100	489,700	496,333
18	565,135	556,340	611,602	577,692

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran

after exclusion
number of laboratories p: 16
general mean y**: 502,283
standard deviation s: 158,217

notes:
Lab. 4 straggler after Cochran Test
Lab. 10 was not able to test at -30 °C

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	58,750	61,400	63,230	61,127
2	63,060	61,610	67,620	64,097
3	22,290	22,700	21,840	22,277
4	62,220	51,171	41,706	51,699
5	32,500	26,100	22,051	26,884
6	70,142	71,327	64,359	68,609
7	71,940	65,810	68,370	68,707
8	61,331	64,117	62,094	62,514
9	55,729	49,447	53,330	52,835
10	48,780	52,120	44,130	48,343
11	59,700	68,340	67,950	65,330
12	43,408	38,091	55,279	45,593
13	51,389	54,658	60,700	55,583
14	104,500	103,100	95,720	101,107
15	46,284	44,051	45,464	45,266
16	50,360	54,620	41,720	48,900
17	57,340	55,440	55,390	56,057
18	56,680	57,068	59,622	57,790

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 55,706
standard deviation s: 17,020

notes:
Lab. 14 straggler after Grubbs Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	213,700	218,700	224,500	218,967
2	215,200	207,000	227,300	216,500
3	65,110	68,260	65,030	66,133
4	209,600	173,741	141,329	174,890
5	106,000	86,900	70,669	87,856
6	205,104	210,169	179,581	198,285
7	244,400	223,900	231,800	233,367
8	224,690	236,622	228,524	229,945
9	214,260	186,460	202,412	201,044
10	164,800	154,900	130,000	149,900
11	204,000	233,700	226,800	221,500
12	173,885	158,655	179,671	170,737
13	175,962	186,094	206,668	189,575
14	308,900	304,400	291,300	301,533
15	162,297	153,986	162,798	159,694
16	167,500	187,700	138,400	164,533
17	200,900	195,100	193,900	196,633
18	179,857	187,846	190,035	185,913

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 187,056
standard deviation s: 53,190

notes:
Lab. 4 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	486,900	496,500	513,400	498,933
2	507,100	482,400	535,000	508,167
3	151,800	161,400	152,000	155,067
4	457,600	384,260	310,819	384,226
5	251,000	210,000	166,211	209,070
6	433,100	441,049	369,091	414,414
7	553,200	524,500	522,000	533,233
8	538,258	565,094	544,135	549,162
9	464,936	398,701	429,151	430,929
10	258,100	251,000	224,100	244,400
11	482,100	549,900	523,400	518,467
12	394,027	368,048	409,497	390,524
13	415,274	429,449	478,872	441,198
14	648,300	635,600	626,300	636,733
15	336,600	323,315	336,208	332,041
16	362,400	417,000	305,200	361,533
17	458,600	446,900	442,700	449,400
18	413,685	424,071	434,863	424,206

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 415,650
standard deviation s: 123,702

notes:
Lab. 4 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	762,300	771,400	807,800	780,500
2	726,000	653,200	714,700	697,967
3	246,200	258,900	245,400	250,167
4	614,100	528,565	422,946	521,870
5	383,000	323,000	247,224	317,741
6	584,540	593,768	493,724	557,344
7	784,500	757,200	740,300	760,667
8	807,546	846,548	809,256	821,117
9	743,694	638,928	686,780	689,801
10				
11	664,100	764,200	729,000	719,100
12	64,928	592,238	61,917	239,694
13	602,079	617,453	680,177	633,236
14	879,000	861,900	851,600	864,167
15	541,057	523,199	547,443	537,233
16	487,000	548,900	416,100	484,000
17	617,000	599,600	596,100	604,233
18	671,813	661,614	732,767	688,731

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 16
general mean y**: 620,492
standard deviation s: 170,991

notes:
Lab. 10 was not able to test at -30 °C

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	70,100	73,160	75,360	72,873
2	74,950	73,010	80,160	76,040
3	27,960	28,580	27,410	27,983
4	73,950	60,800	49,590	61,447
5	38,800	31,600	26,515	32,305
6	81,415	82,818	74,459	79,564
7	85,370	78,070	80,990	81,477
8	73,740	76,820	74,385	74,981
9	67,103	59,626	64,282	63,670
10	58,550	62,380	52,620	57,850
11	71,520	81,720	80,860	78,033
12	51,787	45,790	66,083	54,553
13	61,863	65,490	72,485	66,613
14	121,300	120,000	112,000	117,767
15	55,934	53,339	55,073	54,782
16	60,120	65,260	49,680	58,353
17	68,250	66,060	65,930	66,747
18	67,543	68,261	70,906	68,904

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 66,330
standard deviation s: 19,513

notes:
Lab. 14 straggler after Grubbs Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	237,500	242,800	249,800	243,367
2	239,400	230,200	253,300	240,967
3	77,650	81,540	77,490	78,893
4	231,700	192,324	156,369	193,464
5	119,000	98,500	79,592	99,031
6	225,716	231,167	197,017	217,966
7	270,800	248,900	257,000	258,900
8	252,765	264,595	255,461	257,607
9	240,389	209,669	227,213	225,757
10	185,900	175,500	146,500	169,300
11	229,000	262,100	253,800	248,300
12	193,277	175,639	200,343	189,753
13	198,269	208,684	230,829	212,594
14	338,600	334,300	321,300	331,400
15	183,936	174,911	184,889	181,245
16	186,800	209,400	154,200	183,467
17	223,000	216,700	215,400	218,367
18	201,598	210,531	212,414	208,181

analytical exclusions:
-
statistical exclusions:
-
after exclusion
number of laboratories p: 18
general mean y**: 280,809
standard deviation s: 57,739
notes:
Lab. 4 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	513,100	522,600	541,300	525,667
2	535,400	509,400	564,600	536,467
3	173,000	184,100	173,000	176,700
4	478,600	402,801	325,913	402,438
5	269,000	225,000	178,068	224,023
6	457,050	464,554	375,896	432,500
7	581,900	553,000	549,000	561,300
8	574,942	601,439	578,671	585,017
9	499,499	428,719	461,825	463,348
10	282,400	275,600	245,800	267,933
11	512,400	584,200	555,000	550,533
12	417,472	389,739	433,085	413,432
13	443,016	456,144	507,187	468,782
14	677,100	666,200	657,200	666,833
15	364,432	350,546	365,066	360,014
16	383,400	440,000	322,400	381,933
17	482,200	470,300	465,900	472,800
18	439,863	451,417	460,063	450,447

analytical exclusions:
-
statistical exclusions:
-
after exclusion
number of laboratories p: 18
general mean y**: 441,120
standard deviation s: 127,445
notes:
Lab. 4 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	776,800	786,700	823,900	795,800
2	748,900	673,100	737,000	719,667
3	272,800	287,100	271,800	277,233
4	631,300	542,074	434,235	535,870
5	398,000	337,000	257,488	330,829
6	604,878	613,289	511,179	576,449
7	804,800	778,000	759,100	780,633
8	839,940	875,031	837,064	850,678
9	773,274	665,228	715,931	718,144
10				
11	690,900	793,000	755,400	746,433
12	65,000	603,975	61,974	243,650
13	625,256	639,236	702,824	655,772
14	901,200	884,300	875,900	887,133
15	566,212	549,247	574,560	563,340
16	503,800	567,600	429,800	500,400
17	635,200	618,000	614,100	622,433
18	692,825	682,816	751,018	708,886

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran

after exclusion
number of laboratories p: 16
general mean y**: 641,856
standard deviation s: 172,428

notes:
Lab. 10 was not able to test at -30 °C

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **10 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	129,900	134,800	139,300	134,667
2	137,100	132,400	145,600	138,367
3	62,320	64,370	61,180	62,623
4	133,200	110,640	89,590	111,143
5	73,200	59,700	49,046	60,649
6	137,797	140,044	124,216	134,019
7	154,400	143,400	146,300	148,033
8	138,748	144,345	140,041	141,045
9	128,300	113,765	122,711	121,592
10	111,200	117,000	97,970	108,723
11	134,700	153,600	149,700	146,000
12	96,819	86,763	119,974	101,186
13	118,193	123,112	135,675	125,660
14	204,900	204,300	194,100	201,100
15	107,492	103,420	106,991	105,968
16	111,300	121,800	91,500	108,200
17	125,300	121,900	121,200	122,800
18	123,064	125,458	128,449	125,657

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 122,080
standard deviation s: 31,565

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	339,500	346,300	357,900	347,900
2	345,500	330,600	364,700	346,933
3	142,900	150,600	142,300	145,267
4	322,600	270,406	219,382	270,796
5	179,000	149,000	118,236	148,745
6	313,195	319,950	271,606	301,584
7	384,300	359,300	363,700	369,100
8	372,172	388,525	375,553	378,750
9	357,436	311,455	338,011	335,634
10	278,100	265,800	221,500	255,133
11	339,500	386,800	371,200	365,833
12	277,767	253,172	289,617	273,519
13	296,382	306,697	337,798	313,626
14	462,500	459,200	447,000	456,233
15	278,121	267,246	281,448	275,605
16	268,900	301,900	222,100	264,300
17	317,600	310,200	307,600	311,800
18	294,176	306,996	307,712	302,961

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 303,540
standard deviation s: 75,869

notes:
-

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	606,600	617,100	641,700	621,800
2	640,500	605,200	672,900	639,533
3	268,300	285,300	267,600	273,733
4	551,400	467,231	378,989	465,873
5	333,000	280,000	219,540	277,513
6	543,874	551,335	448,126	514,445
7	684,200	658,200	645,800	662,733
8	699,802	730,428	705,069	711,766
9	629,034	541,934	583,968	584,979
10	383,500	377,600	331,600	364,233
11	626,100	710,000	671,900	669,333
12	496,987	464,476	518,350	493,271
13	546,453	555,077	612,447	571,326
14	782,100	774,400	769,000	775,167
15	469,197	455,338	474,261	466,266
16	459,200	523,500	385,500	456,067
17	567,200	555,500	549,800	557,500
18	533,986	550,126	553,722	545,945

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 536,194
standard deviation s: 138,858

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	825,400	836,000	876,800	846,067
2	826,000	741,200	813,400	793,533
3	380,800	401,900	379,100	387,267
4	674,400	581,263	467,578	574,414
5	446,000	379,000	289,664	371,555
6	675,113	684,160	572,421	643,898
7	871,800	848,800	822,300	847,633
8	930,838	971,209	931,296	944,448
9	874,777	754,029	814,335	814,380
10				
11	782,600	891,200	845,600	839,800
12	58,745	642,155	60,233	253,711
13	706,815	713,038	781,012	733,622
14	975,400	958,800	953,500	962,567
15	651,960	636,380	665,060	651,133
16	561,300	631,600	476,400	556,433
17	697,000	680,600	675,000	684,200
18	744,198	749,651	813,378	769,076

analytical exclusions:

-

statistical exclusions:

Lab. 12 excluded after Cochran Test

after exclusion

number of laboratories p: 16

general mean y**: 713,752

standard deviation s: 175,865

notes:

Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

9.2 Complex shear modulus G* for 25/55-55

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	30,690	32,050	30,910	31,217
2	37,600	36,200	35,040	36,280
3	9,982	9,681	10,100	9,921
4	20,190	24,150	27,600	23,980
5	18,301	15,109	14,582	15,998
6	27,789	30,425	30,632	29,615
7	35,060	33,610	36,750	35,140
8	30,748	32,767	31,887	31,801
9	24,861	25,823	26,837	25,840
10	27,790	30,200	32,240	30,077
11	41,410	39,390	37,030	39,277
12	29,051	37,259	34,544	33,618
13	26,490	25,391	29,589	27,157
14	54,840	49,810	49,700	51,450
15	21,957	22,742	21,709	22,136
16	28,540	29,600	25,650	27,930
17	34,600	36,170	33,210	34,660
18	27,551	26,510	28,011	27,357

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 29,636
standard deviation s: 9,021

notes:
-

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	139,700	144,800	140,400	141,633
2	157,000	148,700	144,400	150,033
3	35,790	33,590	36,850	35,410
4	90,140	103,900	119,800	104,613
5	73,968	61,303	61,842	65,704
6	116,346	125,927	121,424	121,232
7	146,800	141,300	153,400	147,167
8	138,176	148,040	143,357	143,191
9	101,498	116,507	121,444	113,150
10	107,400	129,600	135,300	124,100
11	172,400	165,800	156,800	165,000
12	99,222	157,906	126,740	127,956
13	109,996	106,628	125,076	113,900
14	197,700	180,800	183,000	187,167
15	94,394	92,891	90,355	92,547
16	119,600	123,400	107,200	116,733
17	140,800	144,600	133,500	139,633
18	111,935	111,912	118,125	113,991

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 17
general mean y**: 122,071
standard deviation s: 36,002

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	379,000	392,500	383,200	384,900
2	435,500	410,700	400,200	415,467
3	99,580	92,900	101,300	97,927
4	239,900	273,600	313,700	275,733
5	218,042	182,431	174,708	191,727
6	314,214	336,083	324,964	325,087
7	389,500	380,100	406,500	392,033
8	381,241	401,547	384,944	389,244
9	257,967	275,818	287,158	273,648
10	199,700	226,100	261,200	229,000
11	474,800	451,000	430,500	452,100
12	298,106	415,878	340,994	351,659
13	299,824	294,525	343,508	312,619
14	495,700	460,700	473,600	476,667
15	217,622	223,102	215,386	218,703
16	297,900	305,700	273,200	292,267
17	365,200	369,200	344,600	359,667
18	318,749	296,660	352,179	322,529

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 320,054
standard deviation s: 96,148

notes:
Lab. 12 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	672,700	698,800	685,000	685,500
2	648,700	643,800	627,400	639,967
3	177,400	167,000	178,300	174,233
4	363,000	400,800	464,300	409,367
5	362,286	315,926	297,566	325,259
6	474,511	489,381	487,410	483,768
7	603,000	591,600	630,600	608,400
8	614,677	642,326	622,094	626,366
9	479,480	508,936	515,881	501,432
10				
11	698,100	658,700	639,700	665,500
12	28,484	11,036	586,642	208,721
13	471,503	461,891	526,458	486,617
14	733,800	688,900	724,000	715,567
15	381,185	402,372	385,545	389,701
16	425,200	425,400	398,300	416,300
17	522,800	528,300	495,500	515,533
18	642,272	553,089	650,596	615,319

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 16
general mean y**: 516,177
standard deviation s: 148,194

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	75,230	78,470	75,830	76,510
2	89,010	85,220	82,700	85,643
3	31,550	30,060	31,410	31,007
4	49,090	57,930	66,290	57,770
5	46,750	38,489	37,204	40,814
6	66,745	72,365	71,631	70,247
7	82,610	79,800	86,490	82,967
8	76,163	80,871	78,615	78,549
9	51,433	64,786	67,166	61,128
10	66,190	73,050	77,090	72,110
11	100,600	95,300	90,870	95,590
12	67,880	82,965	78,432	76,425
13	65,654	63,592	73,810	67,685
14	120,400	110,300	111,400	114,033
15	55,447	57,973	56,069	56,496
16	68,610	71,030	62,940	67,527
17	80,210	82,820	76,910	79,980
18	66,789	66,602	69,679	67,690

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 71,232
standard deviation s: 18,849

notes:
-

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	241,900	251,600	243,800	245,767
2	272,800	257,500	250,600	260,300
3	87,860	81,570	88,380	85,937
4	153,800	176,100	202,900	177,600
5	139,744	118,208	114,372	124,108
6	202,778	218,322	208,810	209,970
7	250,400	243,500	262,200	252,033
8	247,098	261,481	253,007	253,862
9	196,115	215,537	223,808	211,820
10	190,300	228,900	236,800	218,667
11	306,400	292,000	280,100	292,833
12	176,646	266,655	217,902	220,401
13	198,431	194,066	225,412	205,970
14	328,500	303,200	311,400	314,367
15	175,488	175,915	172,027	174,477
16	205,900	212,400	189,300	202,533
17	238,700	243,000	226,300	236,000
18	196,668	198,900	209,172	201,580

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 17
general mean y**: 215,754
standard deviation s: 56,274

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	503,800	523,900	510,900	512,867
2	586,900	554,000	541,300	560,733
3	194,200	179,900	193,600	189,233
4	312,800	354,400	406,700	357,967
5	303,528	261,436	253,531	272,832
6	422,861	450,885	433,435	435,727
7	516,800	507,200	539,500	521,167
8	531,928	551,689	532,305	538,641
9	391,102	315,074	423,741	376,639
10	302,900	343,300	387,200	344,467
11	651,600	612,900	592,900	619,133
12	404,038	549,832	452,451	468,774
13	417,463	412,674	477,826	435,988
14	653,300	612,200	637,400	634,300
15	328,237	340,059	328,584	332,293
16	400,100	410,200	374,400	394,900
17	483,500	486,800	455,900	475,400
18	439,758	417,076	468,499	441,778

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 439,602
standard deviation s: 116,945

notes:
-

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	762,200	794,600	776,400	777,733
2	777,900	777,700	757,600	771,067
3	301,500	280,800	297,700	293,333
4	419,600	464,100	533,800	472,500
5	436,758	383,155	372,532	397,482
6	568,218	592,831	581,364	580,804
7	707,600	698,200	741,100	715,633
8	759,452	785,712	755,578	766,914
9	615,871	638,113	647,817	633,934
10				
11	861,300	807,200	791,500	820,000
12	31,750	15,865	673,670	240,428
13	581,102	571,793	651,051	601,315
14	870,600	821,700	869,000	853,767
15	491,661	519,311	504,828	505,267
16	515,400	519,800	489,500	508,233
17	627,400	632,400	593,700	617,833
18	738,578	657,640	768,800	721,673

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 16
general mean y**: 627,343
standard deviation s: 160,888

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	88,020	91,810	88,630	89,487
2	103,600	99,090	96,280	99,657
3	38,720	36,790	38,450	37,987
4	57,360	67,500	77,280	67,380
5	54,811	45,302	44,079	48,064
6	77,867	84,291	83,140	81,766
7	95,990	92,950	100,500	96,480
8	89,719	94,523	91,828	92,023
9	70,399	76,407	79,044	75,283
10	77,150	85,320	89,920	84,130
11	117,600	111,300	106,400	111,767
12	78,384	97,770	89,762	88,639
13	77,018	74,713	86,638	79,456
14	138,500	126,900	128,700	131,367
15	65,405	68,575	66,320	66,767
16	79,850	82,680	73,590	78,707
17	92,940	95,780	89,050	92,590
18	78,427	78,056	81,635	79,373

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 83,384
standard deviation s: 21,326

notes:
-

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	265,300	275,900	267,400	269,533
2	299,800	282,500	275,400	285,900
3	102,600	95,110	102,800	100,170
4	168,000	192,500	221,600	194,033
5	155,107	131,256	127,669	138,011
6	222,735	239,495	228,646	230,292
7	274,000	267,000	287,100	276,033
8	273,989	287,988	278,715	280,231
9	219,485	238,797	247,629	235,304
10	209,600	251,600	259,800	240,333
11	337,900	321,300	308,700	322,633
12	195,592	291,735	243,901	243,743
13	219,281	214,707	249,041	227,676
14	358,200	349,300	340,500	349,333
15	194,967	196,664	192,319	194,650
16	225,400	232,300	208,200	221,967
17	260,900	265,300	247,100	257,767
18	217,726	219,969	231,003	222,899

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 17
general mean y**: 238,045
standard deviation s: 61,206

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	527,600	548,600	535,400	537,200
2	616,400	581,800	568,100	588,767
3	217,000	200,800	215,700	211,167
4	326,300	369,500	423,800	373,200
5	322,301	277,987	269,175	289,821
6	443,474	472,779	453,943	456,732
7	541,200	531,200	564,500	545,633
8	563,707	582,676	561,414	569,266
9	418,784	436,626	452,630	436,013
10	324,300	367,700	413,000	368,333
11	686,100	644,000	625,000	651,700
12	424,364	578,098	474,288	492,250
13	440,547	435,818	504,244	460,203
14	683,100	641,700	668,500	664,433
15	350,858	364,782	353,374	356,338
16	419,100	429,600	393,600	414,100
17	505,800	508,900	476,800	497,167
18	460,977	441,898	495,525	466,133

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 465,470
standard deviation s: 119,442

notes:
Lab. 12 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	777,400	810,800	792,000	793,400
2	800,800	802,200	781,000	794,667
3	328,400	305,500	323,400	319,100
4	427,300	475,500	544,100	482,300
5	453,846	397,318	386,113	412,426
6	585,145	610,358	597,356	597,620
7	726,100	716,400	760,700	734,400
8	788,944	812,340	781,357	794,214
9	640,849	663,383	674,663	659,632
10				
11	890,900	833,400	818,700	847,667
12	26,715	13,453	685,571	241,913
13	600,858	591,756	673,514	622,043
14	894,900	844,900	895,000	878,267
15	512,248	542,183	527,696	527,376
16	530,600	536,300	505,300	524,067
17	645,700	650,500	611,100	635,767
18	757,200	674,377	789,461	740,346

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 16
general mean y**: 647,706
standard deviation s: 162,777

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	152,200	158,600	153,500	154,767
2	176,300	168,500	164,100	169,633
3	79,670	75,000	78,370	77,680
4	97,870	114,000	131,100	114,323
5	96,267	80,767	78,377	85,137
6	133,298	143,518	139,984	138,933
7	162,300	158,000	169,900	163,400
8	156,023	163,779	159,066	159,623
9	122,803	135,399	139,324	132,509
10	131,800	146,800	153,700	144,100
11	203,800	192,000	185,400	193,733
12	132,572	164,453	148,662	148,562
13	134,432	131,374	151,763	139,190
14	225,400	207,800	212,700	215,300
15	115,624	121,834	118,335	118,598
16	136,000	140,600	127,200	134,600
17	156,000	159,500	149,200	154,900
18	134,977	135,212	141,140	137,110

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 143,450
standard deviation s: 33,246

notes:
-

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	362,500	377,700	366,000	368,733
2	412,500	389,200	380,000	393,900
3	175,200	161,700	173,400	170,100
4	226,600	257,800	296,700	260,367
5	220,344	188,543	183,325	197,404
6	305,512	327,689	312,176	315,125
7	372,200	364,400	389,800	375,467
8	382,729	399,734	387,141	389,868
9	315,348	337,687	348,925	333,987
10	290,500	344,400	356,000	330,300
11	470,900	444,300	431,400	448,867
12	274,781	393,466	332,943	333,730
13	306,861	302,387	349,836	319,695
14	481,200	447,800	464,400	464,467
15	276,318	282,733	277,260	278,770
16	305,500	314,300	286,500	302,100
17	352,600	356,900	334,100	347,867
18	303,916	307,478	321,765	311,053

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 17
general mean y**: 329,886
standard deviation s: 77,407

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	614,600	640,000	623,900	626,167
2	723,200	685,400	670,700	693,100
3	315,000	290,800	310,000	305,267
4	374,600	423,100	484,800	427,500
5	388,169	336,949	327,172	350,763
6	519,148	553,348	529,646	534,047
7	629,700	619,500	656,300	635,167
8	673,581	694,663	670,846	679,697
9	522,330	542,084	560,369	541,594
10	407,700	462,600	510,500	460,267
11	813,500	758,200	742,400	771,367
12	499,452	671,228	551,776	574,152
13	525,987	522,685	602,184	550,285
14	792,100	748,700	785,500	775,433
15	435,514	457,818	444,944	446,092
16	488,500	500,600	464,200	484,433
17	587,100	590,100	553,600	576,933
18	536,757	525,911	564,453	542,373

analytical exclusions:
-

statistical exclusions:
-

after exclusion
number of laboratories p: 18
general mean y**: 554,147
standard deviation s: 130,452

notes:
Lab. 12 straggler after Cochran Test

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	830,000	866,800	845,400	847,400
2	883,400	886,700	864,100	878,067
3	434,200	402,400	424,300	420,300
4	461,000	512,500	586,700	520,067
5	506,315	444,732	432,091	461,046
6	643,432	675,184	654,750	657,789
7	790,000	781,000	826,900	799,300
8	876,663	902,108	869,327	882,699
9	727,321	751,873	764,582	747,925
10				
11	995,500	925,200	915,100	945,267
12	27,759	11,775	731,857	257,130
13	668,906	662,065	753,037	694,669
14	978,400	926,500	984,300	963,067
15	582,513	620,740	606,336	603,196
16	585,300	594,400	562,200	580,633
17	709,500	714,500	672,800	698,933
18	809,212	723,968	845,078	792,753

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test

after exclusion
number of laboratories p: 16
general mean y**: 718,319
standard deviation s: 168,266

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

9.3 Phase angle δ for 50/70

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	43.62	43.28	43.47	43.46
2	42.31	41.77	41.71	41.93
3	53.66	54.31	53.51	53.83
4	42.76	42.90	42.68	42.78
5	44.16	44.80	43.14	44.03
6	36.87	36.71	35.60	36.39
7	42.01	42.99	41.77	42.26
8	44.56	44.55	44.58	44.57
9	44.88	45.02	45.32	45.07
10	44.89	44.38	43.40	44.22
11	43.80	43.63	42.73	43.39
12	43.69	43.80	41.69	43.06
13	45.52	44.54	44.31	44.79
14	38.04	37.95	39.06	38.35
15	45.51	46.11	46.28	45.97
16	44.19	44.73	43.27	44.06
17	42.98	43.14	43.00	43.04
18	42.35	42.83	42.05	42.41

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 42.93
standard deviation s: 2.37

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	28.32	28.18	28.47	28.32
2	28.30	27.99	28.08	28.12
3	44.39	44.85	44.05	44.43
4	27.45	27.84	27.65	27.65
5	31.20	31.55	30.56	31.10
6	24.85	24.73	24.36	24.65
7	27.64	29.30	27.48	28.14
8	29.73	30.06	29.89	29.90
9	31.82	31.50	29.48	30.93
10	31.29	32.11	31.35	31.58
11	30.39	30.17	29.46	30.01
12	28.15	28.19	28.10	28.15
13	31.53	30.36	29.97	30.62
14	25.13	25.14	26.03	25.43
15	30.76	32.46	32.70	31.97
16	29.56	29.68	29.16	29.47
17	28.14	28.46	28.27	28.29
18	28.95	29.28	28.40	28.88

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 29.01
standard deviation s: 2.02

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	15.64	15.56	15.85	15.68
2	16.05	15.85	16.02	15.97
3	35.30	35.49	34.92	35.24
4	14.07	14.58	14.63	14.43
5	20.12	20.41	18.66	19.73
6	14.88	14.59	14.62	14.70
7	15.24	16.36	15.27	15.62
8	19.32	17.88	19.44	18.88
9	21.48	22.50	21.90	21.96
10	25.43	25.89	24.52	25.28
11	18.13	17.77	17.38	17.76
12	16.12	16.08	16.23	16.14
13	18.87	17.87	17.28	18.01
14	14.18	14.06	14.59	14.28
15	23.40	24.28	24.04	23.91
16	17.07	16.59	16.77	16.81
17	15.34	15.64	15.48	15.49
18	17.38	18.85	19.13	18.45

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 17.83
standard deviation s: 3.27

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **0.1 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	6.60	6.59	6.71	6.63
2	9.69	9.72	9.89	9.77
3	29.52	29.89	29.22	29.54
4	6.79	7.79	9.58	8.05
5	13.27	12.25	12.97	12.83
6	10.10	9.82	10.11	10.01
7	8.36	8.81	8.37	8.51
8	12.62	12.92	11.74	12.43
9	15.01	15.05	15.21	15.09
10				
11	12.44	11.70	11.47	11.87
12	10.93	6.79	9.76	9.16
13	12.25	11.18	10.75	11.39
14	8.24	8.34	8.67	8.42
15	16.40	16.85	16.83	16.69
16	11.18	11.18	10.66	11.01
17	9.61	9.93	9.96	9.83
18	13.08	12.36	12.57	12.67

analytical exclusions:
-

statistical exclusions:
Lab. 4 and 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 14
general mean y**: 17.83
standard deviation s: 3.27

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	34.99	34.69	34.83	34.84
2	33.97	33.58	33.57	33.71
3	44.57	45.18	44.59	44.78
4	33.92	34.11	33.94	33.99
5	36.01	36.41	35.12	35.85
6	29.58	29.50	28.75	29.28
7	33.59	34.85	33.44	33.96
8	36.16	36.12	36.16	36.14
9	36.92	36.71	36.80	36.81
10	36.18	35.57	34.99	35.58
11	35.59	35.41	34.63	35.21
12	35.51	35.89	33.59	35.00
13	36.67	35.76	35.52	35.98
14	30.36	30.26	31.24	30.62
15	37.19	37.64	37.75	37.53
16	35.14	35.62	34.68	35.15
17	34.32	34.54	34.37	34.41
18	34.20	34.60	33.85	34.22

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 34.60
standard deviation s: 2.05

notes:
Lab. 12 straggler after Cochran Test

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	21.33	21.19	21.47	21.33
2	21.52	21.28	21.43	21.41
3	35.12	35.41	34.95	35.16
4	20.16	20.54	20.48	20.39
5	24.14	24.59	23.72	24.15
6	19.13	19.00	18.76	18.96
7	20.78	22.50	20.71	21.33
8	23.23	23.09	23.13	23.15
9	23.74	23.64	23.60	23.66
10	23.75	24.24	24.14	24.04
11	23.24	23.00	22.45	22.90
12	21.48	21.64	21.52	21.55
13	23.94	22.96	22.64	23.18
14	18.98	18.94	19.60	19.17
15	24.66	25.17	25.01	24.95
16	21.95	22.04	21.85	21.95
17	21.08	21.34	21.21	21.21
18	22.46	22.48	22.11	22.35

analytical exclusions:
-

statistical exclusions:
Lab. 7 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 22.15
standard deviation s: 1.73

notes:
Lab. 13 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	10.71	10.62	10.87	10.73
2	11.14	10.98	11.16	11.09
3	26.39	26.44	26.20	26.34
4	9.26	9.67	9.78	9.57
5	13.80	14.11	13.55	13.82
6	10.63	10.52	10.63	10.59
7	10.41	11.45	10.47	10.78
8	12.87	12.71	12.79	12.79
9	14.82	14.83	14.96	14.87
10	18.71	19.05	18.01	18.59
11	12.63	12.34	12.07	12.35
12	11.25	11.21	11.33	11.26
13	13.22	12.43	12.00	12.55
14	9.81	9.67	10.05	9.84
15	16.06	16.55	16.61	16.41
16	11.58	11.19	11.43	11.40
17	10.43	10.65	10.58	10.55
18	12.71	12.61	12.80	12.71

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 12.35
standard deviation s: 2.42

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.0 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	4.10	4.10	4.19	4.13
2	6.21	6.15	6.28	6.21
3	20.90	21.10	20.79	20.93
4	4.37	4.68	5.06	4.70
5	7.95	8.16	8.16	8.09
6	6.83	6.84	7.08	6.92
7	5.32	5.80	5.31	5.48
8	7.31	7.21	7.39	7.30
9	8.50	8.46	8.69	8.55
10				
11	8.13	7.66	7.41	7.73
12	9.08	4.20	13.34	8.87
13	7.98	7.23	6.97	7.39
14	5.45	5.38	5.57	5.47
15	9.61	10.03	9.96	9.87
16	7.18	7.13	6.82	7.04
17	6.16	6.36	6.25	6.26
18	7.98	7.27	7.44	7.56

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 6.85
standard deviation s: 1.51

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	33.37	33.07	33.22	33.22
2	32.42	32.07	32.07	32.19
3	42.73	43.31	42.77	42.94
4	32.21	32.44	32.29	32.32
5	34.32	34.75	33.54	34.20
6	28.29	28.23	27.56	28.03
7	32.00	33.37	31.88	32.42
8	34.50	34.44	34.48	34.47
9	35.30	35.03	35.11	35.15
10	34.53	33.92	33.42	33.96
11	34.01	33.85	33.09	33.65
12	33.99	34.36	31.99	33.45
13	34.98	34.10	33.88	34.32
14	28.95	28.85	29.73	29.18
15	35.47	35.89	36.01	35.79
16	33.44	33.92	33.06	33.47
17	32.70	32.92	32.75	32.79
18	32.56	32.93	32.24	32.58

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 32.98
standard deviation s: 2.01

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	20.05	19.93	20.20	20.06
2	20.32	20.08	20.24	20.21
3	33.34	33.60	33.20	33.38
4	18.83	19.22	19.17	19.07
5	22.79	23.20	22.38	22.79
6	18.10	17.98	17.73	17.94
7	19.54	21.29	19.48	20.10
8	21.97	21.83	21.87	21.89
9	22.47	22.31	22.33	22.37
10	22.35	23.00	22.81	22.72
11	21.91	21.67	21.15	21.58
12	20.28	20.49	20.29	20.35
13	22.55	21.61	21.32	21.83
14	17.87	17.85	18.50	18.07
15	23.23	23.75	23.59	23.52
16	20.58	20.66	20.52	20.59
17	19.80	20.06	19.92	19.93
18	21.18	21.18	20.84	21.07

analytical exclusions:
-

statistical exclusions:
Lab. 7 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 20.87
standard deviation s: 1.65

notes:
Lab. 13 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	9.88	9.80	10.03	9.90
2	10.32	10.15	10.35	10.27
3	24.75	24.77	24.59	24.70
4	8.45	8.86	9.00	8.77
5	12.78	13.10	12.57	12.81
6	9.93	9.79	10.33	10.02
7	9.60	10.63	9.66	9.96
8	11.96	11.80	11.87	11.87
9	13.80	13.82	13.94	13.85
10	17.51	17.82	16.81	17.38
11	11.68	11.40	11.16	11.41
12	10.37	10.35	10.48	10.40
13	12.23	11.48	11.09	11.60
14	9.05	8.99	9.30	9.11
15	14.96	15.39	15.42	15.26
16	10.66	10.30	10.53	10.50
17	9.60	9.82	9.74	9.72
18	11.75	11.66	11.74	11.72

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 11.45
standard deviation s: 2.28

-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **1.59 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	3.73	3.72	3.81	3.75
2	5.70	5.65	5.78	5.71
3	19.36	19.53	19.28	19.39
4	4.05	4.23	4.49	4.26
5	7.26	7.49	7.43	7.40
6	6.40	6.26	6.54	6.40
7	4.84	5.29	4.85	4.99
8	6.71	6.61	6.77	6.70
9	7.78	7.82	8.00	7.86
10				
11	7.45	6.99	6.77	7.07
12	8.63	3.82	12.29	8.25
13	7.32	6.60	6.35	6.76
14	5.03	4.92	5.13	5.03
15	8.80	9.21	9.14	9.05
16	6.55	6.49	6.22	6.42
17	5.61	5.83	5.69	5.71
18	7.27	6.61	6.82	6.90

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 6.27
standard deviation s: 1.39

notes:
Lab. 13 straggler after Cochran Test
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **10 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	27.31	27.03	27.19	27.18
2	26.57	26.36	26.41	26.45
3	35.64	36.10	35.75	35.83
4	25.98	26.26	26.16	26.14
5	28.59	29.03	27.85	28.49
6	23.51	23.46	22.86	23.28
7	26.17	27.58	26.10	26.62
8	28.39	28.72	28.32	28.48
9	29.55	29.23	29.00	29.26
10	28.33	27.73	27.48	27.85
11	28.02	27.85	27.22	27.70
12	28.34	28.71	26.36	27.80
13	28.71	27.91	27.71	28.11
14	23.77	23.65	24.44	23.95
15	29.29	29.70	29.35	29.45
16	27.04	27.47	26.91	27.14
17	26.59	26.82	26.65	26.69
18	26.39	26.91	26.07	26.46

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 27.08
standard deviation s: 1.69

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	15.42	15.31	15.56	15.43
2	15.68	15.50	15.68	15.62
3	26.63	26.78	26.57	26.66
4	14.14	14.49	14.49	14.38
5	17.65	18.49	17.44	17.86
6	14.40	14.27	14.15	14.27
7	15.03	16.70	15.03	15.59
8	17.31	16.96	17.43	17.23
9	17.73	17.30	17.81	17.61
10	17.26	17.83	17.83	17.64
11	16.96	16.73	16.33	16.67
12	15.86	16.09	15.81	15.92
13	17.51	16.71	16.50	16.91
14	13.90	13.79	14.28	13.99
15	17.79	18.21	17.95	17.98
16	15.60	15.64	15.66	15.63
17	15.10	15.34	15.20	15.21
18	16.51	15.99	15.72	16.07

analytical exclusions:
-

statistical exclusions:
Lab. 7 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 16.15
standard deviation s: 1.32

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	7.07	7.02	7.20	7.10
2	7.28	7.16	7.29	7.24
3	18.71	18.68	18.64	18.68
4	5.89	6.20	6.30	6.13
5	9.17	9.52	9.02	9.24
6	7.50	7.44	7.99	7.64
7	6.89	7.71	6.97	7.19
8	9.06	8.95	8.69	8.90
9	9.96	10.10	10.44	10.17
10	13.07	13.34	12.54	12.98
11	8.35	8.12	7.96	8.14
12	7.60	7.57	7.64	7.60
13	8.87	8.30	8.04	8.40
14	6.69	6.58	6.67	6.65
15	10.81	10.93	11.40	11.05
16	7.54	7.26	7.48	7.43
17	6.75	6.95	6.84	6.85
18	8.05	8.06	8.38	8.16

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 8.29
standard deviation s: 1.75

notes:
-

Characteristic: **phase angle δ**
Material: **50/70**
Frequency: **10 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	2.60	2.58	2.65	2.61
2	3.95	3.92	4.04	3.97
3	13.91	14.01	13.91	13.94
4	2.81	2.94	3.09	2.94
5	4.91	5.04	5.06	5.00
6	4.72	4.71	4.90	4.78
7	3.36	3.69	3.39	3.48
8	5.00	4.97	4.64	4.87
9	5.66	5.49	5.50	5.55
10				
11	5.09	4.76	4.61	4.82
12	11.25	2.69	13.85	9.26
13	5.11	4.61	4.48	4.73
14	3.60	3.48	3.57	3.55
15	5.92	6.21	6.27	6.13
16	4.49	4.44	4.28	4.40
17	3.79	3.96	3.83	3.86
18	4.78	4.44	4.23	4.48

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 4.35
standard deviation s: 0.95

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

9.4 Phase angle δ for 25/55-55

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	39.26	39.23	39.29	39.26
2	37.50	37.40	37.53	37.48
3	49.23	48.49	48.51	48.74
4	38.82	38.34	38.44	38.53
5	39.86	40.71	40.67	40.41
6	38.29	37.92	37.05	37.75
7	37.44	37.75	37.40	37.53
8	39.49	39.15	39.08	39.24
9	40.96	40.44	39.86	40.42
10	37.94	38.48	38.02	38.15
11	38.57	38.49	39.05	38.70
12	36.74	35.71	35.95	36.13
13	39.70	40.07	39.88	39.88
14	34.47	34.70	35.25	34.81
15	39.96	40.59	40.61	40.39
16	38.35	38.35	39.19	38.63
17	36.74	36.14	36.73	36.54
18	38.67	39.29	39.05	39.00

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 38.40
standard deviation s: 1.58

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	24.94	25.03	25.06	25.01
2	24.74	24.72	24.83	24.76
3	39.55	39.04	38.52	39.04
4	24.40	24.18	24.16	24.25
5	27.47	27.58	28.00	27.68
6	25.05	24.85	24.46	24.79
7	24.19	24.57	24.19	24.32
8	26.14	25.83	25.80	25.92
9	29.55	27.46	26.32	27.78
10	25.73	25.27	25.09	25.36
11	25.81	25.47	26.10	25.79
12	25.86	23.32	24.10	24.43
13	26.62	26.94	26.69	26.75
14	22.84	23.18	23.73	23.25
15	26.06	28.59	29.03	27.89
16	24.73	24.69	25.79	25.07
17	23.90	23.44	23.91	23.75
18	26.22	26.43	26.39	26.35

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 25.48
standard deviation s: 1.41

notes:
Lab. 9 straggler after Cochran Test

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	13.56	13.67	13.62	13.62
2	14.02	13.94	14.08	14.01
3	30.36	29.96	29.40	29.91
4	12.71	12.43	12.38	12.51
5	13.65	17.60	17.27	16.17
6	13.97	13.96	13.58	13.84
7	13.41	13.61	13.46	13.49
8	16.67	16.84	16.42	16.64
9	19.08	20.05	19.75	19.63
10	19.30	19.43	18.06	18.93
11	14.83	14.46	15.05	14.78
12	14.22	13.09	13.35	13.55
13	15.57	15.84	15.51	15.64
14	13.05	13.30	13.81	13.39
15	20.47	20.48	20.80	20.58
16	14.00	13.95	14.95	14.30
17	13.21	13.10	13.26	13.19
18	15.57	12.65	17.34	15.19

analytical exclusions:
-

statistical exclusions:
Lab. 5 and 8 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 15
general mean y**: 15.21
standard deviation s: 2.56

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **0.1 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	6.08	6.22	6.09	6.13
2	8.72	8.90	8.93	8.85
3	24.74	24.24	23.90	24.29
4	7.17	7.19	7.37	7.25
5	11.67	12.18	8.71	10.86
6	8.78	9.07	8.50	8.78
7	7.90	8.09	7.91	7.97
8	11.37	9.75	11.64	10.92
9	13.94	13.79	13.80	13.85
10				
11	9.91	9.62	10.27	9.93
12	17.31	16.24	6.45	13.34
13	9.98	10.26	10.28	10.17
14	8.24	8.45	8.76	8.48
15	14.34	14.18	14.87	14.46
16	9.30	9.74	10.01	9.68
17	8.79	8.59	8.81	8.73
18	12.04	12.00	12.68	12.24

analytical exclusions:
-

statistical exclusions:
Lab. 5, 8 and 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 13
general mean y**: 9.73
standard deviation s: 2.46

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	31.21	31.21	31.26	31.23
2	30.01	29.93	30.04	29.99
3	40.40	39.83	39.80	40.01
4	30.73	30.22	30.30	30.42
5	32.25	32.93	32.96	32.71
6	30.54	30.27	29.57	30.13
7	29.80	30.12	29.77	29.90
8	31.87	31.55	31.53	31.65
9	32.55	32.47	32.33	32.45
10	30.34	30.75	30.40	30.50
11	31.04	30.86	31.38	31.09
12	29.61	29.30	28.79	29.23
13	31.67	32.01	31.85	31.84
14	27.50	27.88	28.31	27.90
15	32.62	32.87	33.10	32.86
16	30.39	30.35	31.16	30.63
17	29.28	28.82	29.23	29.11
18	31.14	31.37	31.28	31.26

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 30.76
standard deviation s: 1.34

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	18.64	18.71	18.75	18.70
2	18.80	18.75	18.87	18.81
3	30.98	30.67	30.20	30.62
4	17.97	17.81	17.69	17.82
5	21.17	21.68	21.68	21.51
6	18.85	18.67	18.44	18.65
7	18.17	18.46	18.17	18.27
8	20.14	19.77	19.81	19.91
9	21.67	20.60	20.45	20.91
10	19.38	18.79	18.77	18.98
11	19.61	19.23	19.77	19.54
12	19.96	17.83	18.24	18.68
13	20.09	20.38	20.14	20.20
14	17.39	17.69	18.25	17.78
15	20.85	21.74	21.84	21.48
16	18.30	18.24	19.18	18.57
17	17.88	17.61	17.93	17.81
18	20.02	20.10	19.92	20.01

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 16
general mean y**: 19.31
standard deviation s: 1.25

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	9.47	9.56	9.51	9.51
2	9.95	9.88	9.97	9.93
3	22.51	22.29	21.85	22.22
4	8.67	8.51	8.42	8.54
5	11.97	12.33	12.26	12.19
6	9.81	9.77	9.55	9.71
7	9.37	9.56	9.43	9.45
8	11.33	11.18	11.24	11.25
9	13.92	13.50	13.29	13.57
10	13.96	14.05	12.91	13.64
11	10.48	10.12	10.64	10.41
12	10.08	9.27	9.44	9.60
13	11.00	11.21	10.99	11.07
14	9.37	9.48	9.95	9.60
15	13.60	14.15	14.44	14.06
16	9.65	9.59	10.36	9.87
17	9.23	9.14	9.38	9.25
18	11.79	11.44	12.39	11.87

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 10.80
standard deviation s: 1.71

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.0 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	4.12	4.21	4.12	4.15
2	5.90	6.03	6.05	5.99
3	17.45	17.16	16.91	17.17
4	4.75	4.76	4.74	4.75
5	7.31	7.44	7.41	7.39
6	6.01	6.26	5.72	6.00
7	5.30	5.42	5.29	5.34
8	7.03	6.90	7.01	6.98
9	8.30	8.05	8.19	8.18
10				
11	6.94	6.58	6.96	6.83
12	14.71	14.48	4.26	11.15
13	6.80	7.01	6.99	6.93
14	5.66	5.79	6.00	5.82
15	8.48	8.80	9.01	8.76
16	6.21	6.53	6.72	6.49
17	5.90	5.89	6.05	5.95
18	8.17	7.68	8.11	7.99

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 6.50
standard deviation s: 1.27

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	29.72	29.73	29.78	29.74
2	28.65	28.57	28.67	28.63
3	38.67	38.13	38.09	38.30
4	29.17	28.73	28.79	28.90
5	30.73	31.39	31.40	31.17
6	29.11	28.85	28.19	28.72
7	28.40	28.70	28.37	28.49
8	30.41	30.08	30.07	30.19
9	34.37	31.00	30.87	32.08
10	28.92	29.31	28.98	29.07
11	29.63	29.43	29.95	29.67
12	28.35	27.93	27.56	27.94
13	30.19	30.52	30.36	30.36
14	26.31	26.65	27.15	26.70
15	31.08	31.35	31.56	31.33
16	28.94	28.89	29.68	29.17
17	27.91	27.47	27.86	27.75
18	29.65	29.86	29.77	29.76

analytical exclusions:
-

statistical exclusions:
Lab. 9 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 16
general mean y**: 29.22
standard deviation s: 1.23

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	17.52	17.59	17.63	17.58
2	17.76	17.71	17.82	17.76
3	29.37	29.09	28.64	29.03
4	16.85	16.61	16.52	16.66
5	19.95	20.45	20.45	20.28
6	17.74	17.59	17.36	17.56
7	17.10	17.37	17.10	17.19
8	19.04	18.69	18.72	18.82
9	20.60	19.49	19.33	19.81
10	18.23	17.65	17.66	17.85
11	18.48	18.10	18.63	18.40
12	18.86	16.84	16.98	17.56
13	18.91	19.21	18.96	19.03
14	16.45	16.82	17.19	16.82
15	19.66	20.48	20.57	20.24
16	17.16	17.10	18.01	17.42
17	16.79	16.56	16.87	16.74
18	18.86	18.92	18.75	18.84

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 16
general mean y**: 18.19
standard deviation s: 1.20

notes:
-

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Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	8.81	8.89	8.83	8.84
2	9.28	9.22	9.33	9.28
3	21.09	20.89	20.48	20.82
4	8.02	7.87	7.81	7.90
5	11.14	11.48	11.44	11.36
6	9.12	9.09	8.90	9.04
7	8.72	8.91	8.79	8.81
8	10.60	10.45	10.51	10.52
9	13.01	12.64	12.45	12.70
10	13.04	13.13	12.03	12.73
11	9.74	9.40	9.89	9.68
12	9.39	8.58	8.78	8.92
13	10.24	10.44	10.21	10.30
14	8.81	8.87	9.26	8.98
15	12.66	13.21	13.44	13.10
16	8.94	8.89	9.60	9.14
17	8.57	8.48	8.71	8.59
18	10.88	10.64	11.48	11.00

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 10.05
standard deviation s: 1.61

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **1.59 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	3.82	3.90	3.81	3.84
2	5.51	5.62	5.65	5.59
3	16.18	15.93	15.69	15.93
4	4.44	4.35	4.41	4.40
5	6.76	6.88	6.86	6.83
6	5.55	5.77	5.25	5.52
7	4.91	5.03	4.89	4.94
8	6.55	6.42	6.52	6.50
9	7.71	7.54	7.60	7.62
10				
11	6.41	6.08	6.43	6.31
12	15.38	10.92	3.97	10.09
13	6.28	6.50	6.48	6.42
14	5.29	5.33	5.60	5.41
15	7.84	8.16	8.33	8.11
16	5.75	6.03	6.21	6.00
17	5.45	5.41	5.60	5.49
18	7.61	7.08	7.54	7.41

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 6.03
standard deviation s: 1.18

notes:
Lab. 10 was not able to test at -30 °C

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **0 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	24.18	24.22	24.25	24.22
2	23.54	23.36	23.44	23.45
3	32.08	31.67	31.64	31.80
4	23.57	23.16	23.15	23.29
5	25.45	26.01	26.18	25.88
6	23.79	23.58	23.08	23.48
7	23.20	23.48	23.20	23.29
8	24.96	24.82	24.95	24.91
9	27.64	25.98	25.54	26.38
10	23.60	23.91	23.69	23.73
11	24.30	24.04	24.52	24.29
12	23.46	23.31	22.74	23.17
13	24.69	24.99	24.84	24.84
14	21.67	21.99	22.48	22.05
15	25.46	25.67	25.87	25.67
16	23.43	23.34	24.06	23.61
17	22.73	22.38	22.67	22.59
18	24.13	24.40	24.33	24.29

analytical exclusions:
-

statistical exclusions:
Lab. 9 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 16
general mean y**: 23.92
standard deviation s: 1.04

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-10 °C**

laboratory	sample 1 y_1	sample 2 y_2	sample 3 y_3	cell mean y_i*
	[kPa]	[kPa]	[kPa]	[kPa]
1	13.55	13.62	13.66	13.61
2	13.84	13.76	13.89	13.83
3	23.34	23.19	22.80	23.11
4	12.84	12.65	12.56	12.68
5	15.46	16.09	15.85	15.80
6	13.83	13.69	13.57	13.70
7	13.31	13.56	13.32	13.40
8	14.90	14.57	15.01	14.83
9	16.25	15.71	15.23	15.73
10	14.11	13.63	13.64	13.79
11	14.36	13.98	14.45	14.26
12	14.97	13.35	13.22	13.85
13	14.75	15.00	14.78	14.84
14	12.94	13.19	13.64	13.26
15	14.95	16.10	15.68	15.58
16	13.10	13.04	13.81	13.32
17	12.96	12.78	13.04	12.93
18	14.27	14.58	14.51	14.45

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test
after exclusion
number of laboratories p: 16
general mean y**: 14.13
standard deviation s: 0.99

notes:
-

https://doi.org/10.24355/dbbs.084-202110271159-0

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-20 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	6.59	6.66	6.60	6.62
2	6.82	6.76	6.90	6.83
3	15.96	15.87	15.53	15.79
4	5.91	5.78	5.72	5.80
5	8.56	8.44	8.44	8.48
6	6.88	6.82	6.67	6.79
7	6.52	6.70	6.62	6.61
8	8.43	7.72	8.17	8.10
9	9.74	9.64	9.79	9.73
10	9.80	9.89	9.00	9.56
11	7.20	6.91	7.30	7.14
12	7.09	6.47	6.61	6.72
13	7.69	7.85	7.66	7.73
14	6.74	6.66	7.01	6.80
15	9.03	9.82	9.65	9.50
16	6.59	6.53	7.09	6.74
17	6.29	6.24	6.44	6.32
18	7.72	7.88	8.27	7.96

analytical exclusions:
-

statistical exclusions:
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 17
general mean y**: 7.50
standard deviation s: 1.21

notes:
-

Characteristic: **phase angle δ**
Material: **25/55-55**
Frequency: **10 Hz**
Temperature: **-30 °C**

laboratory	sample 1 y_1 [kPa]	sample 2 y_2 [kPa]	sample 3 y_3 [kPa]	cell mean y_i* [kPa]
1	2.88	2.92	2.86	2.89
2	4.07	4.15	4.26	4.16
3	11.78	11.64	11.46	11.63
4	3.25	3.23	3.24	3.24
5	5.07	5.30	4.94	5.10
6	4.16	4.36	3.96	4.16
7	3.65	3.76	3.66	3.69
8	5.18	4.78	4.83	4.93
9	5.53	5.44	5.75	5.57
10				
11	4.64	4.39	4.64	4.56
12	11.27	13.01	3.00	9.10
13	4.67	4.83	4.80	4.77
14	3.87	3.93	4.14	3.98
15	5.79	5.56	5.95	5.77
16	4.19	4.39	4.53	4.37
17	3.93	3.92	4.05	3.97
18	5.17	4.82	5.32	5.10

analytical exclusions:
-

statistical exclusions:
Lab. 12 excluded after Cochran Test
Lab. 3 excluded after Grubbs Test

after exclusion
number of laboratories p: 15
general mean y**: 4.42
standard deviation s: 0.81

notes:
Lab. 10 was not able to test at -30 °C

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